

# Assessment of the Susceptibility of Coals to Spontaneous Heating using Wet Oxidation Potential Difference Technique



Mithilesh Kumar Rajak, G.K Pradhan, M.J.A.

**Abstract:** This study is proposed to assess the susceptibility of coals from different Indian coal mines and correlation study to find out the suitability of the methods for assessment of spontaneous heating of coals. The wet oxidation potential difference experiments (WOPD) were conducted for 20 different coal samples collected from different India coal mines. The coal samples were collected by channel sampling and prepared according to the Indian Standards. The energy content of all the coal samples in terms of calorific values was measured with the Bomb Calorimeter and proximate analysis was also conducted so as to obtain volatile matter, fixed carbon, moisture content and calorific value. Correlation study was carried out to find the suitability of the method to assess the spontaneous heating susceptibility of coal. Generally, Crossing Point Temperature (CPT) method is used for assessment of spontaneous heating susceptibility of coal. However, this method has certain drawbacks. The results are dependent upon packing density, rate of heating and oxygen flow rate etc., and sometimes results are not reproducible. Moreover, it takes long hours (more than three hours) to complete the experiment. In comparison to CPT, WOPD experiments only takes about 40 minutes for completion and the results are reproducible as well. From the correlation analysis, it was found that WOPD indicated a very fair accuracy for the measure of spontaneous heating susceptibility as it showed high correlation with the intrinsic properties such as moisture and volatile matter.

**Keyword:** Spontaneous heating, Susceptibility, Wet Oxidation Potential Difference, Crossing Point Temperature, Proximate Analysis

## I. INTRODUCTION

The coal mining industry has been suffering perennial losses mainly due to coal fires caused by spontaneous heating. In order to prevent the occurrence of coal mine fires, there have been rigorous works and experiments carried out across the globe. Spontaneous heating occurs due to the rise in temperature of coal stacks to its ignition point where it leads to fire. At this point when coal is presented to air, some of its uncovered surfaces ingest free oxygen at a quickly and oxidation results in the emission of toxic gasses. This happens at encompassing temperatures which prompt open flame. Scientists are constantly endeavoring to prevent such unconstrained heating of coal so as to have an uninterrupted production of coal without incurring significant losses caused by the same. An appropriate

method of different experiments of the spontaneous heating of coal is required, so that production of coal could be done within the incubation period. Different experimental procedures are embraced taking into account the estimation of oxidation rate and ignition temperature, viz., Crossing Point Temperature, Differential Thermal Analysis, Differential Scanning Calorimetry, Russian U-index, Olpinski Index etc. [1]-[3]. It has likewise been seen by different researchers that a portion of these strategies are tedious, monotonous and don't give reproducible outcomes. Keeping these in view, the authors have carried out an extensive study for determining the spontaneous heating tendency of a large number of coal samples by using wet oxidation potential difference technique.

## II. EXISTING METHODS EXPLORED BY RESEARCHERS

Susceptibility to the unconstrained warming of various coals shifts over a wide range and it is imperative to evaluate their level of inclination for taking necessary preventive measures against the event of flames. The susceptibility of spontaneous heating is used to get better knowledge regarding the storage of coal and better methods of extraction so as to obtain maximum extraction within the safe time span. The correlation analysis provides a mathematical approach to the susceptibility of coal to spontaneous heating and helps in identifying the different intrinsic properties on which spontaneous heating depends or the relationship among the obtained results.

Different scientists worked through different approach on susceptibility of coal to the spontaneous heating are mentioned as following:

Scholars contrived a portioned way to examine intersection point temperature bend identified Inflection point, Crossing point and Ignition point as three stages in crossing point temperature curve [4]. They conducted wet oxidation of coal sample in alkaline potassium permanganate (KMnO<sub>4</sub>) solution and analyzed the changes in potential between a soaked calomel cathode and carbon anode inundated in coal oxidant blend inside a timeframe at a steady temperature. They also conducted and found as to how the moisture content of give coal on sample affects the spontaneous ignition of coal and found that the rate of heating of coal maximum if moisture content was ranging in between 7 wt% and 20 wt%.

Scientists conducted Infusion of inert gases techniques to suppress the heating and fire. Application of nitrogen foam, Inertization of Goaf, Water mist etc. Their report included the successful application at different mines across the globe [5].

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Researchers came up with a database of coal selfheating rates in adiabatic conditions at The University of Queensland. Coals from Australia (Queensland and New South Wales), New Zealand (Northland South Island), Indonesia and the United States were tested and found definitive correlations and trends of coal intrinsic properties on spontaneous heating rates [6]. They developed an scheme of propensity rating which was used frequently in Australia. They used a fuzzy expert system and the commonly used artificial neural networks (ANN) for forecasting the self heating of coals [7]. They carried out proximate, ultimate and petrographic analysis on 30 samples of varying ranks from different coalfields of the country. Correlation studies between the intrinsic properties and CPT was carried out to identify the parameters for prediction purpose. Using moisture, volatile matter, and ash content as input parameters, CPT was predicted using fuzzy logic algorithm. Triangular fuzzy membership function was adopted for describing input variables.

Researchers conducted over 600 experiments with more than 50 samples of coal collected from different coal seams (searing and non-blazing) covering diverse geological areas in India and related the oxygen, moisture, volatile matter and hydrogen contents of the coal with the outputs of WOPD method conducted at various experimental conditions [8]. The optimum conditions were found to be 0.2N KMnO<sub>4</sub> solution in 1N KOH at 45°C. As for conclusion, he found that the correlation analysis it was corroborating with field observations and a good correlation was obtained from the CPT carried out on the samples.

### III. EXPERIMENTAL INVESTIGATIONS

20 coal samples were collected from different Indian coal fields. The Intrinsic properties of these samples were determined by carrying out proximate analysis and calorific value. The wet oxidation potential difference experiment was conducted to assess the susceptibility of coals to the spontaneous heating. The details of the experimentation have been presented here.

#### A. Sample Collection and Preparation

In this work, the procedure of channel sampling was followed as laid in IS 436 Part I/Section I - 1964 [9]. The sampling was done in open and exposed place from the roof to the floor. The sample was taken in the form of a channel having dimensions of 30 x 10 cm, i.e., 30 cm in width and 10 cm in depth. First two parallel lines 30 cm apart and almost perpendicular to the bedding planes of the seam were cut. The dirt bands exceeding 10 cm were excluded. The marked sample was then cut to a depth of 10 cm and the coal sample was collected on clean cloth or tarpaulin placed at the bottom so as to prevent the flying of broken pieces during the cutting of sample.

#### B. Proximate Analysis (IS 1350 Part I-1984)

The proximate analysis is done to find out volatile matter, ash content moisture content, and fixed carbon present in the coal sample through experiments in which generally, the coal sample is exposed to heat at different conditions according to the requirement. Procedures for conducting the proximate analysis for different parameters has been stipulated in Indian Standard 1350 (Part-I-1984) and same is followed [10].

#### C. Procedure for determination of Moisture Content(M)

- 1gm air dried coal sample of size 212 $\mu$  is weighed in a silica crucible.
- A pre heated electric hot air oven maintained at 108°C is used for the experiment where the coal sample is kept for a stipulated time of 1.5 hours.
- After the duration of 1.5 hours, the coal sample in the silica crucible is taken out of the oven wearing safety gloves and tong and then it is allowed to cool in a desiccator for 15 minutes before weighing it.
- The weight loss is the moisture which is expressed in percentage.

The moisture content is determined as:-

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

Where,

X = weight of the empty silica crucible (gm)

Y = combined weight of the silica crucible

and coal sample taken in it (gm)

Z = combined weight of the silica crucible and coal sample after heating (gm)

#### D. Procedure for determination of Volatile Matter (VM)

The volatile matter test is carried out in a special silica crucible with the given dimensions. First, the weight of empty silica crucible is noted and then coal sample of around 1g by weight is taken in the crucible. The silica crucible along with the coal sample in it is placed in the muffled furnace which is maintained at 925°C with the crucible being covered by a lid. The coal sample is heated in the furnace for 7 minutes after which it is safely taken out using tongs and then placed in the desiccator to cool down and later, when it cools down, the crucible is again weighed without the lid.

The volatile matter is determined as:

$$\text{Volatile Matter \%} = \frac{Y - Z}{Y - X} * 100$$

Where,

X = weight of empty crucible, g

Y = Combined weight of crucible and coal sample before heating in gram

Z = Combined weight of crucible and coal sample after heating in gram

#### E. Procedure for determination of Ash Content(A)

The silica crucible is pre heated at 800°C for 1 hour to ensure that it is free from any other mineral matter other than the coal. After heating, it is allowed to cool at the room temperature and weighed.

About 1 gm of the prepared coal sample of -212µ size is taken in the cooled crucible and again it is weighed and then it is placed in the muffle furnace which is preheated at 450°C for 30 minutes and then the furnace is set to a raised temperature of 850°C for 1 hour. After 1 hour, the hot crucible is removed from the furnace wearing gloves and using tongs to hold the crucible and then it is placed in the desiccator for 15 minutes to cool down and then it is weighed.

The calculation for ash content is given as:

$$\text{Ash \%} = \frac{Z - X}{Y - X} * 100$$

Where,

X = weight of empty crucible (gm)

Y = Combined weight of coal sample and crucible in grams before heating

Z = Combined weight of coal sample and crucible in grams after heating

**F. Procedure for determination of Fixed Carbon (FC)**

The portion of coal sample excluding the ash, volatile matter and moisture content is known to be the fixed carbon as per definition. The approximated yield of coke from coal is mainly comprised of the fixed carbon and ash content of the coal. Thus, the fixed carbon could be easily determined by the method of subtracting summation of the moisture content, volatile matter and ash content together from 100%.

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

The results of proximate analysis for all the coal samples have been presented in table-I

**Table-I: Results of Proximate Analysis**

Sl. No.	Sample	M (%)	VM (%)	A (%)	FC (%)
1	MCL 1	4.261	30.92	36.71	28.109
2	MCL 2	4.48	30.897	30.58	34.043
3	MCL 3	6.31	28.41	23.62	41.66
4	SECL 1	6.46	28.52	20.13	44.89
5	ECL 1	4.64	28.59	22.18	44.59
6	SECL 2	2.22	31.75	36.79	29.24
7	BCCL 1	0.93	21.5	23.92	53.65
8	CCL 1	0.86	26.2	20.09	52.85
9	BCCL 2	0.93	20.68	27.45	50.94
10	ECL 2	0.9	25.09	21.05	52.96
11	MCL 4	6.74	26.25	15.56	51.45
12	ECL 3	0.71	22.84	15.54	60.91
13	MCL 5	11.53	33.52	28.23	26.72
14	CCL 2	5.68	28.78	21.53	44.01
15	CCL 3	6.48	32.12	16.51	44.89
16	SCCL 1	3.57	34.99	6.19	55.25
17	SCCL 2	3.06	28.49	25.26	43.19

18	NCL 1	6.15	36.15	7.99	49.71
19	NECL 1	1.57	41.08	5.88	51.47
20	MCL 6	5.82	24.85	27.31	42.02

**G. Procedure for determination of calorific value**

A weighed amount of coal sample between 0.5g to 1g is placed in the silica crucible. The crucible is supported over the ring. A fine magnesium wire is stretched acrossed the electrodes touching the coal sample. Oxygen is supplied into the bomb until 25-30 atmospheric pressured is reached. The water jacket is filled with 2ltrs of water. The bomb is placed inside water jacket and manually the weight of the sample is entered and then process starts. The current is switched on the fuel in the crucible burns with the evolution of heat. The heat generated by the burning of coal is transferred to water which is stirred during whole experiment by the electric stirrer. Once the bomb fires, it gives a high pitch sound. It gives the calorific value automatically after the coal sample is completely burned.

**Table-II: Calorific Values of coal samples**

Sample	Calorific Value (Kcal/kg)
MCL 1	6335.58
MCL 2	4737.61
MCL 3	5114.38
SECL 1	6080.33
ECL 1	6641.64
SECL 2	4932.65
BCCL 1	6352.23
CCL 1	6634.47
BCCL 2	5963.30
ECL 2	6528.48
MCL 4	6312.08
ECL 3	5370.17
MCL 5	5554.18
CCL 2	5029.71
CCL 3	5760.03
SCCL 1	6810.03
SCCL 2	5262.16
NCL 1	6137.47
NECL 1	7861.22
MCL 6	5104.93

**IV. WET OXIDATION POTENTIAL DIFFERENCE METHOD**

In the alkaline permanganate solution, coal oxidation occurs and the concentration of magnetite ion increases compared to the permanganate ion and thus it leads to the potential difference. The potential difference change is measured by the carbon electrode sank in the alkaline permanganate solution. The change in concentration occurs due to the chemical reaction of coal with the KOH and KMnO4 solution.

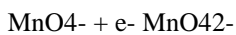
The KMnO4 and KOH solution with the coal sample forms an electrochemical cell which is constantly stirred by an electromagnetic magnetic stirrer to generate EMF against a standard potential of 0.56 V. A graph is plotted between EMF and time which gives an idea about the spontaneous combustion of coal

**A. Principle of Wet oxidation**

**Wet Oxidation Potential Difference Apparatus**



An alkaline potassium permanganate (KMnO4) solution is prepared and used for the wet oxidation potential experiment. The reduction of permanganate ion to magnetite ion is represented as follows:



The standard electrode potential (E0) for the redox reaction of the permanganate ion is 0.56V. The electrode potential can be calculated by the following equation:

$$E = E_0 - \frac{RT}{F} \ln \left( \frac{[\text{MnO}_4^{2-}]}{[\text{MnO}_4^-]} \right)$$

Where,

R = Universal gas constant

T = Temperature

F = Faraday’s constant

The addition of coal leads to the oxidation of the coal sample while the permanganate ion is reduced to magnetite ion which results in the change in EMF and the change in potential occurs till the whole oxidation is completely over. The change in potential of the carbon electrode is measured for taking the results. The electrode is represented as carbon/MnO2-.

**B. Procedure**

At first, 100ml of a deci-normal solution of potassium permanganate (KMnO4) in 1N potassium hydroxide (KOH) solution is prepared in a glass beaker, and then it is placed on the magnetic stirrer machine along with the Teflon coated fish of the magnetic stirrer. After that, the carbon electrode is fixed as a cathode and calomel as an anode. The magnetic stirrer and the wet oxidation apparatus is switched on. Then about 0.5 gm of coal sample of (-212µ size) weighed and added thoroughly into the alkaline solution. The magnetic stirrer produces a homogeneous mixture and is controlled with the rotation accordingly. The readings are noted down from a voltmeter at an interval of 1 minute till the potential difference tends to become constant.

The graphs are plotted between Time and Potential differences obtained for all the coal samples and have been presented in Figures 1 to 4. It could be observed from the graphs that the potential difference for each sample becomes constant after 30 minutes. Thus, 30 minutes was selected as the time interval for the comparison of the potential difference values. The results of potential difference values after 30 minutes for all the coal samples have been presented in Table-III.

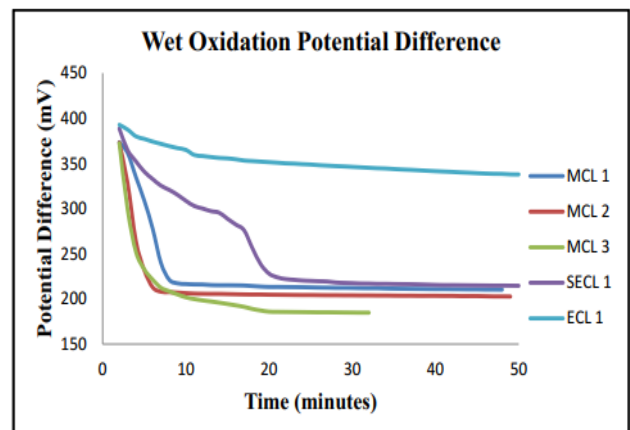


Fig. 1. Wet Oxidation Potential curve (Sample 1 – 5)

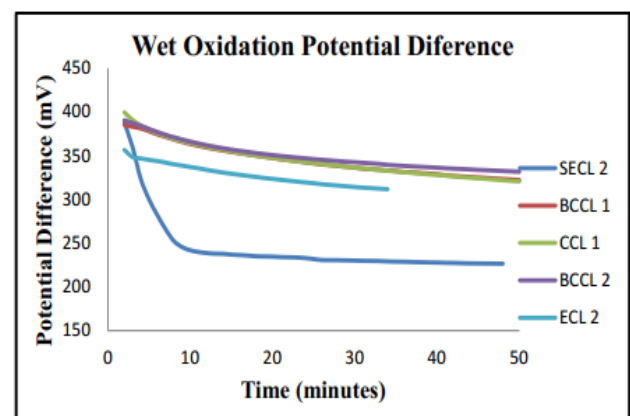


Fig. 2. Wet Oxidation Potential curve (Sample 6 – 10)

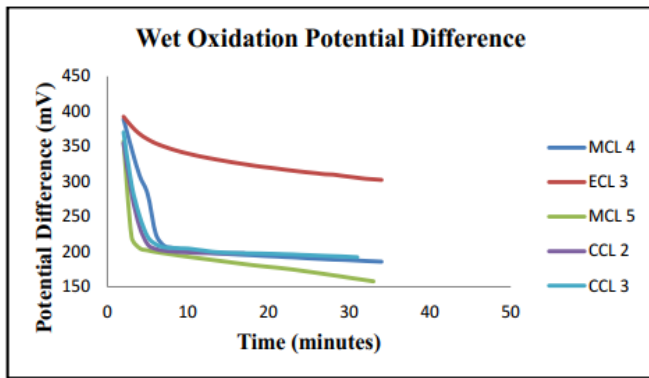


Fig. 3. Wet Oxidation Potential curve (Sample 11 – 15)

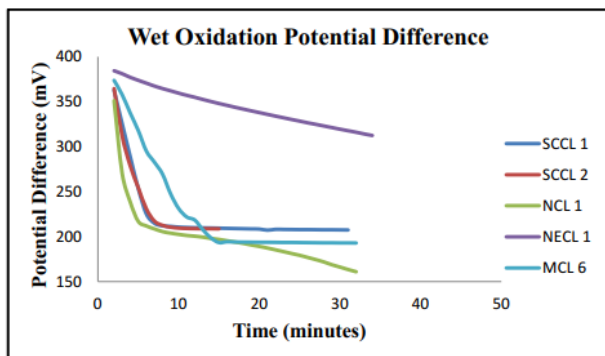


Fig. 4. Wet Oxidation Potential curve (Sample 16 – 20)

Table-III: Results of the wet oxidation potential difference

Sample	WOPD after 30 minutes
MCL 1	161.8
MCL 2	169.3
MCL 3	187.6
SECL 1	171.7
ECL 1	47.6
SECL 2	157.9
BCCL 1	50.7
CCL 1	65
BCCL 2	49.3
ECL 2	43.8
MCL 4	202
ECL 3	88.5
MCL 5	198
CCL 2	157.1
CCL 3	178.2
SCCL 1	155.5
SCCL 2	156
NCL 1	190.3
NECL 1	68.4
MCL 6	180.9

## V. DISCUSSION

The evaluation of 20 samples for the intrinsic properties as well as their wet oxidation potential difference is carried out. Of 20 samples, 6 samples were from MCL, 2 samples from SECL, 3 samples from ECL, 2 samples from BCCL, 3 samples from CCL, 2 samples from SCCL and 1 sample each from NCL and NECL. The sample collection was done following the Indian Standards. The determination of intrinsic parameters was done conducting Proximate Analysis whereas the spontaneous heating susceptibility was measured by the Wet Oxidation Potential Difference. The Gross Calorific Value was also determined by the Bomb Calorimeter.

The moisture content of the coal samples varied from 0.71% (ECL 3) to 11.53% (MCL 5). It was observed that the coal samples from MCL had higher moisture content were more susceptible to spontaneous heating.

The volatile matter of the coal samples varied from 20.68% (BCCL 2) to 41.08% (NECL 1). It was observed that coal samples from BCCL had the lowest volatile matter than others. The SCCL and SECL coal samples had considerably high volatile matter content than that of MCL and CCL.

The ash content of the coal samples varied between 5.88% (NECL 1) to 36.79% (SECL 2). The coal samples from MCL showed relatively higher average ash content. The fixed carbon content varied between 26.73% (MCL 5) to 60.90% (ECL 3).

The gross calorific values varied between 4737.615 Kcal/kg (MCL 2) to 7861.22 Kcal/kg (NECL 1). The coal samples from CCL showed considerably high calorific values.

In order to establish the effects of intrinsic properties on susceptibility to spontaneous combustion of coal samples, correlation studies were conducted. The correlation coefficients between the intrinsic properties and susceptibility indices were found out by taking the parameters determined by proximate analysis viz. moisture, volatile matter, ash; and gross calorific value as independent variables and the susceptibility indices determined by wet oxidation potential analyses as a dependent variable. Empirical relations were also devised between them and respective curves were plotted on x-y axes having taken independent variables on horizontal axis and dependent variables on vertical axis. The correlation plots have been presented in Figure 5, 6, 7, 8, 9, and the empirical relationship and correlation coefficient between WOPD value and the intrinsic properties of the samples are presented in table-IV.

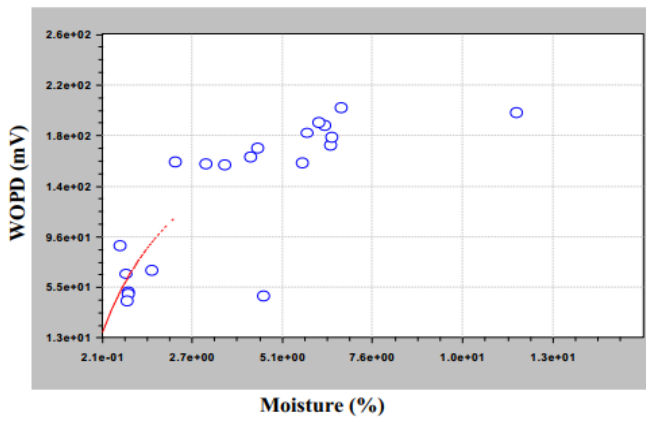


Fig. 5. Correlation plot between WOPD and Moisture content

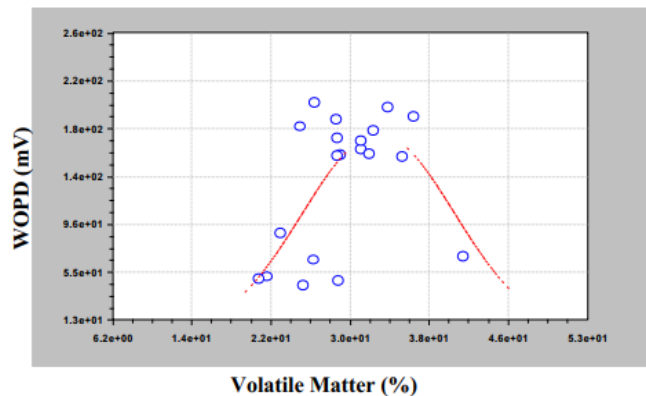


Fig. 6. Correlation plot between WOPD and volatile matter

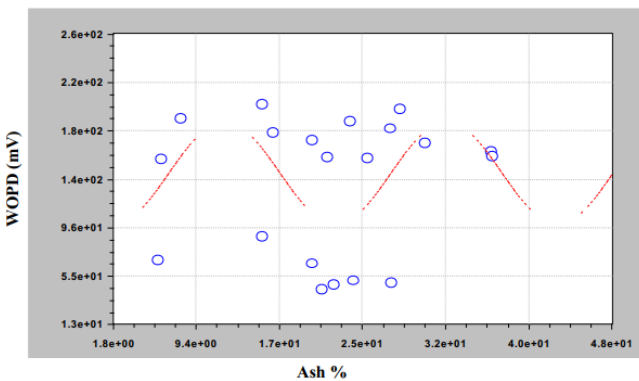


Fig. 7. Correlation plot between WOPD and Ash content

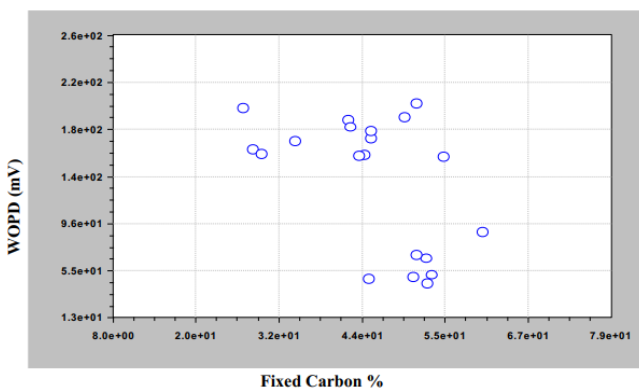


Fig. 8. Correlation plot between WOPD and Fixed Carbon content

Table-IV: Correlation between Wet Oxidation Potential Difference (WOPD) and Intrinsic Properties

S.No	Independent variable	Empirical relation	Correlation Coefficient
1	M	$WOPD = \frac{248.168}{2.634 + M}$	0.84
2	VM	$WOPD = 174.45e^{-\frac{(VM - 32.55)^2}{2(7.58)^2}}$	0.67
3	A	$WOPD = 143.337 + 43.501\cos(0.306A + 2.613)$	0.4
4	FC	$WOPD = 278.132 - 3.194FC$	0.52
5	GCV	$WOPD = 355.243 - 3.194FC$	0.50

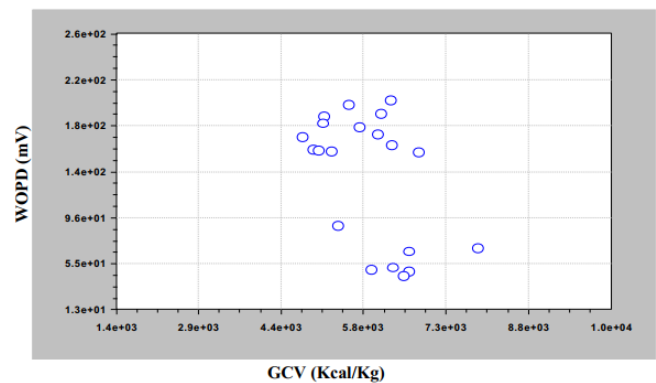


Fig. 9. Correlation plot between WOPD and Gross Calorific Value

It can be noted that Volatile Matter and Moisture values showed a good correlation whereas other intrinsic properties showed low values. Thus, they are not so reliable for the assessment of to the susceptibility to spontaneous heating.

WOPD showed correlation values nearly 0.5 with gross calorific value and fixed carbon. It is clear from the correlation plots that the wet oxidation potential difference after 30 minutes is lower for coals having high fixed carbon and high calorific value, which means they are of higher rank. This corroborates the findings of earlier research (Tarfadar, Ray et al ), this method may not be an optimum measure of susceptibility to spontaneous heating. Hence, this method could be an alternative method for The assessment of spontaneous heating of coal.

The wet oxidation potential values varied between 43.8 mV (ECL 2) to 202 mV (MCL 4).

The coal samples having WOPD values less than 100 mV were ECL 1, BCCL 1, CCL 1, BCCL 2, ECL 2 & NECL 1 indicating that they are poorly susceptible to spontaneous heating. The coal samples MCL 1, MCL 2, MCL 3, SECL 1, SECL 2, MCL 4, MCL 5, CCL 2, CCL 3, SCCL 1, SCCL 2, NCL 1 & MCL 6 were highly susceptible as they had WOPD values greater than 120 mV.

## VI. CONCLUSION

In India, mostly crossing point temperature (CPT) method is used for assessment of susceptibility of coal to spontaneous heating. However, this method has certain drawbacks. The results are the functions of packing density, heating rate and flow rate of oxygen etc., and sometimes these results are not reproducible. Moreover it takes more than three hours to complete the experiment. In this study, WOPD method was adopted for the evaluation of spontaneous heating of coal. In comparison to crossing point temperature, the WOPD experiment takes only nearly 40 minutes for completion. The WOPD method provides excellent repeatability of the results for the same sample, but in case of crossing point temperature; the repeatability of the experimental results is not so good as that of WOPD method. The correlation analysis between potential difference and intrinsic properties was found to be fairly accurate, which indicates that this method should be considered as a measure of susceptibility to spontaneous heating. Moreover, the wet oxidation method is very handy and easy to perform compared to CPT. Hence, WOPD method can be considered a more useful method for the assessment of susceptibility of coal to spontaneous heating.

From the above discussion, it may be concluded that the coal samples from MCL, SCCL and SECL had extreme values in the WOPD experiments which indicates that it is highly susceptible to spontaneous heating. Among other samples, CCL 2, CCL 3 and NCL 1 are also highly susceptible to spontaneous heating.

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