Development of a petrographic technique to assess the spontaneous combustion susceptibility of Indian coals

Niroj Kumar Mohalik\textsuperscript{a,b}, Edward Lester\textsuperscript{b}, and Ian Stuarts Lowndes\textsuperscript{b}

\textsuperscript{a}Mine Ventilation Division, CSIR-CIMFR, Dhanbad, India; \textsuperscript{b}Process and Environmental Research Division Faculty of Engineering, University of Nottingham, Nottingham, UK

ABSTRACT
Petrographic studies are commonly used to categorize the potential utilization of coals. Eleven coal samples from the Jharia coalfield (JCF), India, were studied using petrographic techniques to investigate maceral content, reflectance, and textural characteristics. Multiple test samples of each coal were slowly oxidized under controlled laboratory conditions from an ambient temperature of 30°C to 300°C to investigate the morphology of oxidized coals. The petrographic characterization of coals before and after oxidation showed significant changes in both morphology and vitrinite reflectance. The oxidation of coal particles produced three predominant textural changes: particles with homogeneous change of reflectance (HC\textsubscript{v}), particles with oxidation rims (OR\textsubscript{v}), and particles with no changes were observed (U\textsubscript{v}), respectively. These textural characteristics were used to indicate how particles had interacted with oxygen at low temperatures during the early stages of oxidation. The morphological classification developed provides an alternative method to confirm the susceptibility of a coal to spontaneous combustion. Conventional thermal parameters such as crossing point temperature (CPT) were unable to identify the coals prone to spontaneous combustion. However, certain petrographic parameters could be combined with CPT values to provide a much more accurate measure for susceptibility to spontaneous combustion.

Introduction
Spontaneous combustion of coal is a very complex physicochemical reaction caused by several 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) and extrinsic factors (particle size, geological condition, mining methods, and environmental condition). The interaction of each agent is not straightforward, but oxidation seems to be responsible for the observed compositional and structural changes. Different countries have adopted various methods to assess the propensity of coal to spontaneous combustion in the laboratory (Mohanlil 2013; Nimaje and Tripathy 2016). The different methods proposed by previous researchers may be cataloged under three types: a determination of the chemical constituents, oxygen avidity studies, and thermal studies (Banerjee 2000). These test methods

CONTACT
Niroj Kumar Mohalik\textsuperscript{a} niroj.mohalik@gmail.com Principal Scientist, Mine Ventilation Division, CSIR-Central Institute of Mining and Fuel Research, Barwa Road, Dhanbad, Jharkhand - 826015 India

Color versions of one or more of the figures in the article can be found online at www.tandfonline.com/gcop.

© 2017 Taylor & Francis Group, LLC
may be reclassified into five categories: chemical compositional studies, microscopy studies, thermal studies, oxygen avidity studies, and advanced miscellaneous techniques (Mohalik 2013). Petrographic studies identify maceral composition and vitrinite reflectance and can also be used to identify the mineral content and the textural relationships within the coal samples. Geologists conclude that counts of maceral with vitrinite reflectance are useful to determine the susceptibility of coals to spontaneous combustion (Avila, Wu, and Lester 2014; Benfell, Beamish, and Rodgers 1997; Chandra et al. 1991; Chandra and Prasad 1990; Mohalik 2013; Morris and Atkinson 1988). The total amount of vitrinite plus liptinite influences the susceptibility of a coal to spontaneous combustion, and the risks of spontaneous combustion decrease with increase in coalification (Avila 2012; Chandra et al. 1991; Chandra and Prasad 1990; Misra and Singh 1994). The rate of oxidation is directly proportional to the vitrinite content and inversely proportional to the higher the rank of coal (Kruszewska and Du Cann 1996; Kruszewska, Labuschagne, and Du Cann 1996; Pattanaik, Behera, and Singh 2011). The studies of Ogunsola and Mukula (Ogunsola and Mikula 1990) conclude that the spontaneous combustion characteristics of Nigerian coals are coal specific and not simply rank dependent. All major macerals groups (vitrinite, liptinite, and inertinite) are susceptible to weathering and oxidation with respect to time, temperature, and environment settings. Among all the macerals vitrinite is the most vulnerable, whereas the inertinite and liptinite groups are more resistant to oxidation (Chandra 1958, 1962; Ingram and Rimstidt 1984; Ivanova and Zaitseva 2006; Marchioni 1983). Both artificial oxidation and natural weathering may cause changes to the petrographic textures of coal. The changes observed in the petrographic texture of an oxidized coal may be due to the reaction temperature and chemical changes, which may be determined by a study of the changes observed in reflectance and morphology of the samples (Avila 2012; Benedict and Berry 1964; Calemma et al. 1995; Gray, Rhoades, and King 1976; Marchioni 1983; Nandi, Ciavaglia, and Montgomery 1977; Pearson and Creaney 1981). Artificial oxidation due to heating presents as a brightening of the vitrinite particle and at the boundaries of particles (oxidation rims) whereas natural weathering processes produce dull rims (Avila 2012; Calemma et al. 1995; Chandra 1958, 1965, 1975; Chen-Brauchler et al. 2009; Crelling, Schader, and Benedict 1979; Gray, Rhoades, and King 1976; Lowenhaupt and Gray 1980; Marchioni 1983; Ndaji and Thomas 1995; Pearson and Creaney 1981; Stach et al. 1982).

The literature reveals the mechanisms of oxidation of a coal either by weathering or artificial oxidation and it is suggested that the study of the artificial oxidation of coals may provide a measure as to the propensivity of these coals to spontaneous combustion. This article outlines the experimental studies performed to provide a petrographic characterization of 11 coal samples collected from mines within the Jharia Coalfield (JCF), Eastern India, some of which are known to be prone to spontaneous combustion. These studies identify the macerals, vitrinite reflectance, and morphological characteristics of fresh and artificial oxidized coals to assess the susceptibility of these coals to spontaneous combustion.

**Materials and methods**

**Sample collection and preparation**

Eleven coal samples were collected from different collieries within the JCF, Dhanbad, India, using channel- and chip-sampling method (Peters 1978) to avoid the weathered
exposed coal surface (IS-436-Part-1/Sec-1 1964). The representative samples were prepared in the laboratory and placed in air-tight containers to minimize oxidation in the presence of air. The coal samples collected were from a range of ranks and were from both fiery and non-fiery classified coal seams. These coal seam fire classifications are based on the historical record of fires as well as the present conditions of the coal seams. The location and geotechnical detail of all of the coal samples are detailed in Table 1 and Figure 1. Among these 11 samples there were 5 samples (sample number: 1, 2, 3, 4, and 5) which have a present or recent past history of spontaneous combustion/fires, the remaining are classified as non-fiery.

Coal characterization studies

Proximate analyses, (including moisture (M), ash (A), volatile matter on dry ash free (daf) basis (VM_{daf}), and fixed carbon (FC)); ultimate analysis (carbon (C_{daf}), hydrogen (H_{daf}), nitrogen (N_{daf}), sulphur (S_{daf}), and oxygen (O_{daf})); fuel ratio (FR), and gross calorific value (GCV) of all coal samples were performed to the ASTM standard. Five individual samples of each coal sample type were tested and the mean data for the repeated tests is presented in Table 2. The crossing point temperature (CPT) method is an accepted standard method for Indian coal mines to determine the susceptibility of coal to spontaneous combustion by the Directorate General Mine Safety (DGMS) (DGMS Cir.Tech.3/1975 1975). In short, the higher the CPT, the lower the susceptibility of the coal to spontaneous combustion. A spontaneous combustion test rig developed at the University of Nottingham (UoN) is used to determine the crossing point temperature (CPT_u) of the coal samples (Avila 2012). The results of the proximate analyses, CPT, and ignition point temperature (IPT) for each coal sample are summarized in Table 2.

Petrographic studies of fresh coals

The determination of the maceral contents and rank of a coal sample requires the preparation and analysis of representative blocks for each coal sample. Polished blocks of each of the coal samples were prepared (Avila, Wu, and Lester 2014) and automated examined using a Leitz Ortholux II POL-BK microscope to perform maceral (BS-ISO-7404-3 2009) and rank analysis (BS-6127-5 1995) of the blocks. Subsequently, the whole

### Table 1. Location, geotechnical details and fire status of collected coal samples.

| Sample Number | Seam Name | Colliery Name | Stratigraphic | Seam Depth (m) | Seam Thickness (m) | Fire Status (Present condition) |
|---------------|-----------|---------------|---------------|----------------|--------------------|---------------------------------|
| 1             | S–14      | Chasnala      | Barakar Series | 80             | 16.16              | Fires exist                     |
| 2             | S–14      | Jitpur        | Barren measure | 440            | 9.07               | Fires exist                     |
| 3             | S–13      | Chasnala      | Barakar Series | 150            | 8.23               | Fires exist                     |
| 4             | S–16      | Jitpur        | Barren Measure | 270            | 4.57               | Fires exist                     |
| 5             | S–11      | Enna          | Barakar Series | 60             | 7.31               | Fires exist                     |
| 6             | S–11      | Bhalgara      | Barakar Series | 95             | 5.80               | No fires                        |
| 7             | S–11      | Simlabahal    | Barakar Series | 230            | 7.70               | No Fires                        |
| 8             | S–12      | Simlabahal    | Barakar Series | 225            | 4.65               | No fires                        |
| 9             | S–10      | Bhalgara      | Barakar Series | 150            | 6.75               | No fires                        |
| 10            | S–10      | Simlabahal    | Barakar Series | 295            | 4.82               | No fires                        |
| 11            | S–09      | Simlabahal    | Barakar Series | 300            | 3.92               | No fires                        |
coal reflectance was measured using image analysis technique with an automatic stage controller on the microscope, using black and white imaging Zeiss AxioCam. The camera images were calibrated using a sapphire standard with a reflectance value of 0.58%. Four mosaic images of 100 frames each (total of 12,012,000 pixels from 10 x 10 individual images) were captured across each coal block and averaged to give an accurate value for each coal sample (Avila 2012; Cloke and Lester 1994; Cloke et al. 1995; Lester et al. 1994). The results of the maceral and rank analysis conducted for all of the 11 coal samples using manual microscopic method and automated image analysis are summarized in Table 3.

Manually point counting derived data has been split into the main macerals but dividing the inertinite macerals into semifusinite and fusinite – representing the main inertinite submacerals. The manual data has the suffix m is then presented as VL_m, SF_m, F_m, and VR_m which means the vitrinite and liptinite (the most reactive fractions), semifusinite, fusinite, and vitrinite reflectance, respectively. The automated image analysis data has the suffix i and is presented as VL_i, SF_i, F_i, and VR_i. It should be noted that the VR_i data do not correspond directly to the VR_o because it does not sample vitrinite in the same way as the manual ISO standard which specifically measures the most homogeneous desmocollinite (normally) and away from any edges. With VR_i, the calculation is based on the average reflectance value was calculated from the main grey scale histogram peak using Eqn. 1:

$$R_{avg} (%) = \frac{\sum_{i=m1}^{i=m2} R_i \times Pix_i (%)}{Pix_i (%) \times N}$$  \hspace{1cm} (1)

Where i - the grey scale value; m1- minima prior to peak maxima; m2- minima post main peak; R - correspondent reflectance value; Pix (%) - pixel percentage value associated; and N- number of grey scale value used. As such, the automated program is not specifically only measuring the best vitrinite but can also have some semifusinite and some liptinite,
Table 2. Proximate, ultimate, GCV, FR, CPT, IPT, and CPT, analysis of the 11 coal samples.

| Sample | Moisture (%wt) | Ash (%wt) | Volatile Matter (%wt daf) | Fixed Carbon (%wt daf) | C (%wt daf) | H (%wt daf) | N (%wt daf) | S (%wt daf) | O (%wt daf) | Calorific Value (MJ/kg) | Fuel Ratio | Crossing Point Temperature (°C) | Ignition Point Temperature (°C) | Crossing Point Temperature (Nottingham) CPT, (°C) |
|--------|---------------|-----------|---------------------------|------------------------|-------------|-------------|-------------|-------------|-------------|---------------------------|------------|---------------------------------|-----------------------------|----------------------------------|
| 1      | 1.14          | 10.31     | 30.69                     | 61.37                  | 77.16       | 4.92        | 1.52        | 0.28        | 16.10       | 29.64                     | 2.26       | 145                            | 164                         | 213                                    |
| 2      | 1.18          | 10.84     | 29.54                     | 61.99                  | 79.77       | 4.81        | 1.58        | 0.27        | 13.57       | 28.86                     | 2.38       | 142                            | 173                         | 206                                    |
| 3      | 1.16          | 7.78      | 32.44                     | 61.52                  | 79.85       | 5.28        | 1.57        | 0.31        | 12.98       | 29.29                     | 2.08       | 136                            | 157                         | 196                                    |
| 4      | 1.30          | 9.34      | 30.06                     | 62.50                  | 79.12       | 4.91        | 1.43        | 0.32        | 14.21       | 28.68                     | 2.33       | 152                            | 186                         | 211                                    |
| 5      | 0.61          | 20.94     | 26.95                     | 57.31                  | 81.31       | 5.02        | 1.62        | 0.00        | 12.06       | 25.59                     | 2.71       | 169                            | 199                         | 231                                    |
| 6      | 1.22          | 16.71     | 28.26                     | 58.88                  | 83.96       | 5.06        | 1.88        | 0.54        | 8.57        | 27.42                     | 2.54       | 171                            | 199                         | 228                                    |
| 7      | 1.25          | 13.40     | 27.22                     | 62.11                  | 82.10       | 4.69        | 1.73        | 0.41        | 11.07       | 28.34                     | 2.67       | 159                            | 185                         | 218                                    |
| 8      | 1.13          | 16.74     | 26.42                     | 60.44                  | 79.99       | 4.81        | 1.64        | 0.50        | 13.05       | 27.24                     | 2.79       | 152                            | 176                         | 211                                    |
| 9      | 0.64          | 17.19     | 24.86                     | 61.74                  | 83.94       | 4.73        | 1.90        | 0.35        | 9.08        | 26.72                     | 3.02       | 165                            | 198                         | 216                                    |
| 10     | 0.63          | 16.75     | 25.08                     | 61.90                  | 84.14       | 4.95        | 1.90        | 0.39        | 8.63        | 27.46                     | 2.99       | 168                            | 197                         | 222                                    |
| 11     | 0.92          | 16.88     | 27.02                     | 59.99                  | 83.53       | 4.94        | 2.00        | 0.50        | 9.04        | 26.77                     | 2.70       | 162                            | 188                         | 223                                    |
Table 3. Petrographic analyses (maceral and rank) of coal samples using the manual and image analysis techniques.

| Sample Number | Manual Analysis | Automated Analysis |
|---------------|-----------------|--------------------|
|               | Vitrinite (Vm)  | Liptinite (Lm)     |
| 1             | 67.2            | 2.4                |
| 2             | 69.2            | 1.6                |
| 3             | 62.4            | 1.6                |
| 4             | 72.0            | 2.8                |
| 5             | 40.4            | 0.8                |
| 6             | 74.4            | 3.2                |
| 7             | 55.2            | 1.2                |
| 8             | 74.8            | 5.6                |
| 9             | 68.4            | 1.2                |
| 10            | 57.2            | 1.2                |
| 11            | 64.8            | 2.0                |
|               | Semifusinite (SFm) | Fusinite (Fm)     |
| 1             | 21.6            | 3.6                |
| 2             | 18.8            | 3.6                |
| 3             | 10.4            | 9.2                |
| 4             | 23.6            | 6.8                |
| 5             | 27.2            | 6.0                |
| 6             | 26.4            | 4.0                |
| 7             | 27.2            | 8.8                |
| 8             | 36.0            | 7.6                |
| 9             | 46.0            | 12.8               |
| 10            | 32.0            | 9.6                |
| 11            | 25.2            | 4.0                |
|               | Vitrine+Liptinite (VLm) | Vitrine Reflectance (VRm) |
| 1             | 74.8            | 0.74               |
| 2             | 77.6            | 0.76               |
| 3             | 80.4            | 0.87               |
| 4             | 69.6            | 0.97               |
| 5             | 66.8            | 1.00               |
| 6             | 69.6            | 1.02               |
| 7             | 64.0            | 1.15               |
| 8             | 56.4            | 1.08               |
| 9             | 41.2            | 1.04               |
| 10            | 58.4            | 1.05               |
| 11            | 70.8            | 1.09               |
|               | Vitrine+Liptinite (VLI) | Semifusinite (SFi) | Fusinite (FI) | Peak Reflectance (VRI) |
| 1             | 74.0            | 22.9               | 3.2           | 0.66               |
| 2             | 78.5            | 19.7               | 4.3           | 0.66               |
| 3             | 79.9            | 10.9               | 9.2           | 0.68               |
| 4             | 71.5            | 21.4               | 7.1           | 0.67               |
| 5             | 66.1            | 26.9               | 1.0           | 0.77               |
| 6             | 72.8            | 22.9               | 4.3           | 0.91               |
| 7             | 65.7            | 18.1               | 16.3          | 0.90               |
| 8             | 58.0            | 37.8               | 4.3           | 0.80               |
| 9             | 41.4            | 45.5               | 13.1          | 0.94               |
| 10            | 60.0            | 21.7               | 18.3          | 1.00               |
| 11            | 73.2            | 24.2               | 2.6           | 0.77               |
where in for maceral reflectance profiles (between macerals) overlap. There are also edge effects that introduce wider reflectance range. However, this parameter is still worth using as it is a relative measure, contains $10^6$ more measurements than manual vitrinite reflectance and VR$_i$ can be compared easily with the VR$_i$ from each treated sample directly.

**Petrographic studies of oxidized coals**

Previous investigations have studied the changes in the vitrinite reflectance and morphology of coal samples heated in the laboratory. These methods have number of inherent limitations: all samples may not be uniformly heated in the sample holder and small particle sizes may produce cracks and binding problems. Given these challenges, an alternative method was explored to study thermally treated coal samples. Freshly prepared coal samples (amount: 10 g, size: +425 micron to −1 mm) were placed in a crucible and heated at 3°C min$^{-1}$ in a muffle furnace up to a maximum temperature of 300°C and held for a further 30 min (200°C and 250°C were tested but did not create any change in reflectance and morphology). Repeatability of experimental results was verified with all coal samples by carrying out five experiments on each coal sample. Manual vitrinite reflectance measurements were taken at the center of any vitrinite particles, to avoid the subjective influence of any oxidation rims surrounding the particle that exhibit no change in reflectance due to heating. Similarly morphology studies of the two morphotypes of vitrinite were considered, i.e., unaltered material (U$_v$) and altered material (A$_v$). The altered material may be grouped into two classes: i.e., the homogenous changes of reflectance (across the whole particle either bright or dull) and the oxidation rims (with homogenous change or not, oxidation rim with cracks, micro fractures, and internal oxidation lines). Changes in the vitrinite morphology were manually determined and identified the presence of the three morphotype groups – unaltered vitrinite (U$_v$), homogenous change of vitrinite (HC$_v$), and vitrinite having oxidation rims (OR$_v$). The mosaic images and their corresponding vitrinite subclassifications U$_v$, HC$_v$, and OR$_v$ are shown in Figure 2(a–c). The vitrinite reflectance measurements were performed on the sample blocks using the automated image analysis techniques. The vitrinite reflectance and morphological results obtained from the oxidized (thermally altered) coals for both manual and image analysis techniques are given in Table 4. Examples of comparison of the analysis of the fresh and oxidized samples using cumulative average techniques for image analysis is shown in Figure 3(a, b). Figure 3(a, b, e) clearly indicate the vitrinite levels of the oxidized sample are very high which means the reflectance values increase after oxidation. But an analysis of Figure 3(c, d, f) reveals that the reflectance of the oxidized coal is lower than the corresponding untreated samples. The observed changes in the reflectance results for both manual and image analysis techniques are given in Figure 4.

**Analysis of results**

**Basic coal characterization**

The proximate analyses of coal samples 1 and 2 (same seam but different depth and stratigraphic stages) are similar in nature even though the difference in depth is very
In the case of samples 5, 6, and 7 (same seam and stratigraphic stages but different depth) the percentage of FC and VM$_{daf}$ increases with depth, whereas the ash and moisture decreases with depth. Similarly, samples 9 and 10 (same seam and stratigraphic stages but different depth) are similar in nature even though the difference in depth is relatively high. The ultimate analysis results for samples 1 and 2 shows that the percentage of C$_{daf}$ and N$_{daf}$ increases with depth and the percentage of H$_{daf}$, S$_{daf}$, and O$_{daf}$ decreases with depth and stratigraphic stages. The percentage of C$_{daf}$ increases for sample number 5, 6, and 7 as depth increases, whereas the percentage of H$_{daf}$, S$_{daf}$, N$_{daf}$, and O$_{daf}$ are similar except in sample 6. The composition of C$_{daf}$, H$_{daf}$, N$_{daf}$, and S$_{daf}$ slightly increases or are similar in nature as depth increases,
whereas oxygen content decreases with depth. The CPTs vary across a range from 136°C in sample 3 to 171°C in sample 6. The CPT values determined for samples 3 is low (<140°C) which is categorized as highly prone to spontaneous combustion, whereas sample number 1, 2, 4, 7, and 8 are in the range of 140°C to 160°C, which are moderately susceptible and sample number 5, 6, 9, 10, and 11 are high (>160°C), which signifies low susceptibility to spontaneous combustion. The determined IPTs vary across a range from 157°C (sample 3) to 171°C (samples 5 and 6). The CPT values vary across a range from between 196°C (sample 8) to 231°C (sample 5).

**Petrographic studies of fresh and oxidized coal**

The vitrinite and liptinite percentages (VL$_{\text{m}}$) show a broad range between 41% and 80% . Semifusinite (SF$_{\text{m}}$) also shows a broad range from 10% to 46%. Fusinite levels (F$_{\text{m}}$) show a more modest range from 3.6% to 12.8%. There were no significant pyrite content levels detected in the coal samples which removes the possibility of self-heating through pyrite based mechanisms(Arisoy and Beamish 2015; Deng et al. 2015). Automated analysis data shows similar ranges. Fresh samples show a VR$_{\text{m}}$ from 0.74% to 1.15%. The vitrinite content increases as depth increases for samples 1 and 2 and sample 9 and 10. Similarly, the rank of coal sample increases as depth increases for all samples. The VRi range is slightly lower for reasons discussed in Section 2.3.

With the oxidized samples, the unaltered vitrinite (U$_{\text{v}}$) percentage varies from 4.8% (sample 3, Table 4) to 49.6% (sample 9: Table 4). The homogenous change vitrinite (HC$_{\text{v}}$) percentage varies from 5.2% (sample 3) to 20.4% (sample 4) whereas oxidation-rim vitrinite (OR$_{\text{v}}$) percentage varies from 90.0% (sample 3) to 34.4% (sample 9), respectively. The oxidation rims around the vitrinite are due to heat exchange between coal and oxygen available at the surface. Samples 1, 2, and 3 are the most reactive as most of the vitrinite is altered (> 70% with HC$_{\text{v}}$ + OR$_{\text{v}}$) and the recorded morphological changes make a significant contribution to the classification of these coals as to their susceptibility to spontaneous heating.
The relative change in vitrinite reflectance from fresh to oxidized (VR\textsubscript{m0}-VR\textsubscript{m}/VR\textsubscript{m}) varies from 0.91% (sample 3) to 1.10% (sample 11) (Figure 4). VR\textsubscript{m} and VR\textsubscript{i} of samples 1, 2, 3, 4, and 5 increases significantly, which means these coals are the most responsive to change during oxidation. Samples 6, 10, and 11 only show a modest increase in reflectance.

**Figure 3.** Reflectance study of fresh and oxidized coal samples (mosaic techniques).
and coal samples 7, 8, and 9 appear to show a modest decrease. Previous studies have suggested that the vitrinite reflectance of samples undergoing artificial oxidation increases (Kus and Misz-Kennan 2017; Misz-Kennan and Fabiańska 2011; Ribeiro et al. 2016). Oxidation of coal under laboratory conditions may also produce a dull appearance (Kus and Misz-Kennan 2017; Misz, Fabiańska, and Ćmiel 2007; Misz-Kennan and Fabianska 2010; Ribeiro et al. 2016). The reason behind the observed reduction in reflectance is not fully understood and needs further study using a large number of samples.

**Chemometric analysis**

**Correlation analysis**

The proximate and petrographic data obtained from the above studies were subsequently statistically compared using correlation analysis, multivariate analysis (i.e., principal component and classification analysis (PCCA), and fixed nonlinear regression models (FNRM)). The Statistica 7.1 statistical package was used (Rencher 2002; STATISTICA-7.0 2004) to perform correlation studies to identify potential relationships between the different spontaneous combustion susceptibilities indices (CPT, CPT_u, U_v, HCV (%), OR_v (%), VR_{mo-m}, and VR_{io-i}); the coal characteristic data provided by the proximate, ultimate, GCV, and FR; and petrographic analyses of coal samples. The values of the correlation coefficients determined ($p < 0.05$ confidence interval) for the above studies are presented in Tables 5 and 6. It reveals that there is a stronger correlation between the oxidized coal properties and the chemical constituents of the coals than for any of the other parameters. A study of the data presented in Tables 5 and 6 reveals that CPT and CPT_u, possesses the highest significance with the ash content ($r = 0.858$, $r = 0.833$), U_v correlates well to VM_{daf} ($r = 0.912$), and VR_{io-i} with VL_m ($r = 0.858$). The positive correlation coefficients reveals it has positive correlation, whereas negative correlation indicates a weak relationship.

**Principal component and classification analysis**

The PCCA technique is widely applied to analyse highly complex datasets. The method seeks to reduce the dimensionality of the data set and to identify relationships between
Table 5. The results of the PCCA correlation study performed between the proximate, ultimate, and susceptibility indices determined for the coal samples (GCV, FR, CPT, CPT$_u$, U$_V$, HC$_V$, OR$_V$, VR$_{mo-m}$, R$_{im-i}$).

|        | M  | Ash | VM$_{daf}$ | FC | C$_{daf}$ | H$_{daf}$ | N$_{daf}$ | S$_{daf}$ | O$_{daf}$ | GCV | FR | CPT | IPT | CPT$_u$ | U$_V$ | HC$_V$ | OR$_V$ | VR$_{mo-m}$ | VR$_{io-i}$ |
|--------|----|-----|-----------|----|-----------|-----------|-----------|-----------|-----------|-----|----|-----|-----|--------|------|-------|-------|------------|------------|
| M      | 1.000 |     |           |    |           |           |           |           |           |     |    |     |     |        |      |       |       |            |            |
| Ash    | -0.700 | 1.000 |           |    |           |           |           |           |           |     |    |     |     |        |      |       |       |            |            |
| VM$_{daf}$ | 0.673 | -0.840 | 1.000 |    |           |           |           |           |           |     |    |     |     |        |      |       |       |            |            |
| FC     | 0.334 | -0.693 | 0.193 | 1.000 |           |           |           |           |           |     |    |     |     |        |      |       |       |            |            |
| C$_{daf}$ | -0.526 | 0.662 | -0.728 | -0.233 | 1.000 |           |           |           |           |     |    |     |     |        |      |       |       |            |            |
| H$_{daf}$ | 0.013 | -0.205 | 0.552 | -0.350 | -0.100 | 1.000 |           |           |           |     |    |     |     |        |      |       |       |            |            |
| N$_{daf}$ | -0.499 | 0.630 | -0.699 | -0.210 | 0.916 | -0.141 | 1.000 |           |           |     |    |     |     |        |      |       |       |            |            |
| S$_{daf}$ | 0.368 | -0.023 | -0.190 | 0.256 | 0.373 | -0.181 | 0.512 | 1.000 |           |     |    |     |     |        |      |       |       |            |            |
| O$_{daf}$ | 0.494 | -0.636 | 0.691 | 0.236 | -0.996 | 0.047 | -0.931 | -0.425 | 1.000 |     |    |     |     |        |      |       |       |            |            |
| GCV    | 0.711 | -0.950 | 0.776 | 0.687 | -0.657 | 0.121 | -0.567 | 0.103 | 0.628 | 1.000 |     |    |     |     |        |      |       |       |            |            |
| FR     | -0.704 | 0.822 | -0.997 | -0.162 | 0.733 | -0.527 | 0.704 | 0.183 | -0.698 | -0.763 | 1.000 |     |    |     |     |        |      |       |       |            |            |
| CPT    | -0.570 | 0.858 | -0.787 | -0.508 | 0.796 | -0.191 | 0.689 | 0.111 | -0.772 | -0.806 | 0.768 | 1.000 |     |    |     |     |        |      |       |       |            |            |
| IPT    | -0.555 | 0.791 | -0.777 | -0.397 | 0.776 | -0.268 | 0.601 | 0.038 | -0.737 | -0.792 | 0.758 | 0.958 | 1.000 |     |    |     |     |        |      |       |       |            |            |
| CPT$_u$ | -0.479 | 0.833 | -0.636 | -0.661 | 0.590 | -0.150 | 0.546 | -0.028 | -0.567 | -0.744 | 0.598 | 0.925 | 0.847 | 1.000 |     |    |     |     |        |      |       |       |            |            |
| U$_V$  | -0.668 | 0.608 | -0.912 | 0.119 | 0.677 | -0.560 | 0.586 | 0.110 | -0.632 | -0.588 | 0.928 | 0.680 | 0.726 | 0.455 | 1.000 |     |    |     |     |        |      |       |       |            |            |
| HC$_V$ | 0.289 | -0.162 | 0.047 | 0.338 | -0.165 | -0.467 | -0.214 | 0.184 | 0.061 | 0.034 | -0.011 | 0.150 | -0.105 | 0.156 | 1.000 |     |    |     |     |        |      |       |       |            |            |
| OR$_V$ | 0.499 | -0.480 | 0.820 | -0.224 | -0.546 | 0.641 | -0.439 | -0.159 | 0.498 | 0.500 | -0.830 | -0.604 | -0.701 | -0.371 | -0.944 | -0.470 | 1.000 |     |    |     |     |        |      |       |       |            |            |
| VR$_{mo-m}$ | 0.094 | -0.458 | 0.550 | 0.135 | -0.539 | 0.243 | -0.390 | -0.361 | 0.526 | 0.522 | -0.545 | -0.405 | -0.391 | -0.200 | -0.484 | -0.015 | 0.436 | 1.000 |     |    |     |     |        |      |       |       |            |            |
| VR$_{io-i}$ | 0.366 | -0.610 | 0.795 | 0.068 | -0.634 | 0.479 | -0.643 | -0.456 | 0.622 | 0.572 | -0.804 | -0.606 | -0.539 | -0.387 | -0.746 | -0.213 | 0.722 | 0.718 | 1.000 |     |    |     |     |        |      |       |       |            |            |
Table 6. The results of the PCCA correlation study performed between the petrographic and susceptibility parameters determined for each of the coal samples (CPT_u, U_V, HC_V, OR_V, VR_m-m, R_m).

|       | VL_m | SF_m | F_m | VR_m | VL_io | SF_io | F_io | VR_io | CPT | IPT | CPT_u | U_V | HC_V | OR_V | VR_m-m | VR_io-i |
|-------|------|------|-----|------|-------|-------|------|-------|-----|-----|-------|-----|------|------|--------|--------|
| VL_m  | 1.000|      |     |      |       |       |      |       |     |     |       |     |      |      |        |        |
| SF_m  | -0.973| 1.000|     |      |       |       |      |       |     |     |       |     |      |      |        |        |
| F_m   | -0.707| 0.525| 1.000|      |       |       |      |       |     |     |       |     |      |      |        |        |
| VR_m  | -0.598| 0.578| 0.433| 1.000|       |       |      |       |     |     |       |     |      |      |        |        |
| VL_io | 0.993| -0.956| -0.733| -0.533| 1.000|       |      |       |     |     |       |     |      |      |        |        |
| SF_io | -0.853| 0.904| 0.375| 0.334| -0.859| 1.000|       |      |     |     |       |     |      |      |        |        |
| F_io  | -0.461| 0.306| 0.764| 0.385| -0.450| -0.036| 1.000|       |     |     |       |     |      |      |        |        |
| VR_io | -0.725| 0.697| 0.538| 0.714| -0.679| 0.379| 0.648| 1.000|     |     |       |     |      |      |        |        |
| CPT   | -0.369| 0.637| 0.147| 0.689| -0.520| 0.407| 0.168| 0.779| 1.000|     |       |     |      |      |        |        |
| IPT   | -0.595| 0.653| 0.193| 0.643| -0.544| 0.434| 0.190| 0.718| 0.958| 1.000|       |     |      |      |        |        |
| CPT_u | -0.322| 0.446| -0.180| 0.516| -0.281| 0.272| -0.100| 0.554| 0.925| 0.847| 1.000|     |     |      |        |        |
| U_V   | -0.880| 0.847| 0.651| 0.662| -0.859| 0.589| 0.630| 0.804| 0.680| 0.726| 0.455| 1.000|     |      |        |        |
| HC_V  | -0.343| 0.382| 0.092| 0.078| -0.312| 0.473| -0.084| -0.138| -0.011| 0.150| -0.105| 0.156| 1.000|     |        |        |
| OR_V  | 0.888| -0.868| -0.614| -0.619| 0.859| -0.662| -0.543| -0.666| -0.604| -0.701| -0.371| -0.944| -0.470| 1.000|       |        |
| VR_m-m | 0.510| -0.447| -0.512| -0.887| 0.460| -0.284| -0.355| -0.562| -0.405| -0.391| -0.200| -0.484| -0.015| 0.436| 1.000|        |
| VR_io-i | 0.876| -0.858| -0.604| -0.816| 0.843| -0.708| -0.417| -0.775| -0.606| -0.539| -0.387| -0.746| -0.213| 0.722| 0.718| 1.000|
variables. An application of these methods to the results of the coal analyses described above concludes that there is low correlation between the results of the ultimate analysis, e.g., nitrogen and low sulfur content (<0.54%) with the tendency of a coal to spontaneously combust. Similarly, from the petrographic analyses it is concluded that the recorded semifusinite (SF$_m$, SF$_i$) and fusinite (F$_m$, F$_i$) levels do not influence the propensity of a coal to spontaneously combustion (Kus and Misz-Kennan 2017; Misz-Kennan and Fabianska 2010; Prakash and Sokol 2015). The Statistica 7.1 computer package was used to perform the PCCA studies. The PCCA analyses performed considered the relationships between the following 11 determined coal characteristic variables (moisture M, ash A, volatile matter VM$_{daf}$, fixed carbon FC, carbon C$_{daf}$, hydrogen H$_{daf}$, oxygen O$_{daf}$, calorific value GCV, fuel ratio FR, vitrinite + liptinite VL$_m$, and vitrinite reflectance VR$_m$), with the four susceptibility indices determined for each coal sample (CPT, CPT$_u$, U$_v$, and VR$_{io-i}$). For this study the principal components (PCs) with eigenvalues greater than 1.0 were considered. However, the total variance for the given data sets, is observed to vary by up to 88% for the first three PCs, and found very small values for the remaining eight PCs (Table 7). The eigenvalues of these three PCs, modify the magnitude of the corresponding eigenvectors significantly (Table 8). The eigenvectors with the largest eigenvalues identify the parameters with the strongest correlation in the data set. Similarly, the scree plot finds the factorial loadings where the observed decrease in eigenvalues appears to level off to the right of the plot (Figure 5). As a result, the first three PCs were selected for the principal component matrix. The factorial loadings and their projections of variable on the factor plane (1x2) are depicted in Table 8 and Figure 6. Factorial loadings close to 1 indicate stronger correlations (Table 8). The projection of the first two factorial loading plots indicate whether the parameters are correlated or not. If the plotted variables are close to the center, it means that some information may be carried over to other axes. The projection of the variables on the factor plane (1 and 2) shows that first group, i.e., A, C$_{daf}$, VR$_m$, VR$_i$, and FR are far from center and but close to each other. Similarly second group (GCV, M, O$_{daf}$, VM$_{daf}$, VL$_m$, and VL$_i$) are on the opposite side of the center as well as to first group. These observations are replicated for each of susceptibility indices (CPT, CPT$_u$, U$_v$, and VR$_{io-i}$).

An analysis of a plot of the weighted parameter will indicate a significant correlation where these parameters are spatially grouped together. The parameters which are in proximity as well as away from center can be grouped into one as they are positively correlated each other. If first group are on opposite sides of the second group then, they are negatively correlated each other. However, variables like fixed carbon and H$_{daf}$ are orthogonal to variable CPT, CPT$_u$, U$_v$, and VR$_{io-i}$, which signifies no correlation. The projections of the cases (for samples) on the factor plane (1x2) are shown in Figure 7. Figure 7 shows that all samples are classified into four clusters with first cluster (X, Y) having samples 1, 2, and 4; second cluster (X, -Y) having sample 3; third cluster (-X, Y) having samples 7, 8, 9, and 10, and fourth cluster (-X, -Y) having samples 5, 6, and 11. All four clusters may divided into three categories as per their susceptibility toward spontaneous tendency, i.e., low (first cluster: samples 1, 2, and 4), medium (second and third cluster: samples 3, 7, 8, 9, and 10) and high (fourth cluster: samples 5, 6, and 11).
Table 7. The eigenvalues of the correlation matrix derived by the PCCA method, and the variance in correlation computed for the four spontaneous combustion susceptibility indices.

| PC | CPT Eigenvalue | % Total variance | Cumulative % | CPTu Eigenvalue | % Total variance | Cumulative % | Uv Eigenvalue | % Total variance | Cumulative % | VRio-i Eigenvalue | % Total variance | Cumulative % |
|----|----------------|------------------|--------------|----------------|----------------|--------------|--------------|----------------|--------------|-----------------|----------------|-------------|
| 1  | 8.88           | 63.42            |              | 8.61           | 61.50          |              | 8.92         | 63.75          |              | 8.84            | 63.17          |              |
| 2  | 2.18           | 15.58            |              | 2.30           | 16.45          |              | 2.24         | 15.97          |              | 2.19            | 15.65          |              |
| 3  | 1.22           | 8.73             |              | 1.22           | 8.72           |              | 1.21         | 8.67           |              | 1.22            | 8.73           |              |
| 4  | 0.77           | 5.50             |              | 0.84           | 6.00           |              | 0.78         | 5.54           |              | 0.79            | 5.63           |              |
| 5  | 0.39           | 2.75             |              | 0.45           | 3.19           |              | 0.38         | 2.71           |              | 0.48            | 3.42           |              |
| 6  | 0.25           | 1.81             |              | 0.26           | 1.85           |              | 0.25         | 1.76           |              | 0.23            | 1.63           |              |
| 7  | 0.15           | 1.09             |              | 0.17           | 1.25           |              | 0.15         | 1.08           |              | 0.15            | 1.06           |              |
| 8  | 0.11           | 0.79             |              | 0.10           | 0.73           |              | 0.04         | 0.31           |              | 0.06            | 0.43           |              |
| 9  | 0.04           | 0.26             |              | 0.03           | 0.24           |              | 0.02         | 0.18           |              | 0.04            | 0.27           |              |
| 10 | 0.01           | 0.07             |              | 0.01           | 0.08           |              | 0.00         | 0.03           |              | 0.00            | 0.01           |              |
Table 8. The computed PCCA factor loadings of the variables in the principal component matrix for the three principal components.

| Variables | PC1 | PC1 | PC1 | PC1 | PC1 | PC2 | PC3 | PC2 | PC3 | PC2 | PC3 | PC2 | PC3 |
|-----------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| M         | 0.706 | 0.180 | 0.343 | 0.710 | 0.160 | -0.276 | 0.711 | 0.236 | 0.317 | 0.683 | 0.286 | -0.340 |
| A         | -0.887 | -0.330 | -0.265 | -0.898 | -0.341 | 0.240 | -0.852 | -0.413 | -0.252 | -0.858 | -0.401 | 0.248 |
| VMdaf     | 0.960 | -0.184 | 0.106 | 0.961 | -0.183 | -0.104 | 0.972 | -0.099 | 0.101 | 0.965 | -0.100 | -0.119 |
| FC        | 0.332 | 0.833 | 0.322 | 0.349 | 0.852 | -0.281 | 0.251 | 0.872 | 0.305 | 0.275 | 0.847 | -0.273 |
| Cdf       | -0.841 | -0.159 | 0.467 | -0.834 | -0.116 | -0.498 | -0.823 | -0.219 | 0.482 | -0.824 | -0.209 | -0.471 |
| Hdaf      | 0.365 | -0.743 | 0.204 | 0.369 | -0.695 | -0.284 | 0.415 | -0.704 | 0.231 | 0.407 | -0.712 | -0.253 |
| Odaf      | 0.812 | 0.187 | -0.507 | 0.804 | 0.142 | 0.541 | 0.791 | 0.247 | -0.523 | 0.795 | 0.232 | 0.515 |
| GCV       | 0.852 | 0.376 | 0.271 | 0.860 | 0.373 | -0.230 | 0.820 | 0.457 | 0.253 | 0.824 | 0.450 | -0.246 |
| FR        | -0.960 | 0.202 | -0.098 | -0.961 | 0.207 | 0.090 | -0.977 | 0.115 | -0.092 | -0.968 | 0.115 | 0.111 |
| VLm       | 0.819 | -0.463 | 0.173 | 0.811 | -0.480 | -0.171 | 0.857 | -0.380 | 0.173 | 0.858 | -0.397 | -0.187 |
| VRm       | -0.774 | 0.038 | 0.299 | -0.770 | 0.057 | -0.295 | -0.769 | -0.028 | 0.301 | -0.789 | 0.021 | -0.313 |
| VLi       | 0.784 | -0.465 | 0.250 | 0.777 | -0.482 | -0.242 | 0.825 | -0.383 | 0.248 | 0.825 | -0.395 | -0.263 |
| VRi       | -0.865 | 0.133 | 0.360 | -0.857 | 0.162 | -0.365 | -0.867 | 0.071 | 0.364 | -0.869 | 0.087 | -0.350 |
| CPT       | -0.883 | -0.259 | 0.085 | -0.723 | -0.457 | 0.090 | -0.908 | 0.336 | 0.002 | 0.865 | -0.282 | 0.090 |
| CPTu      | 0.706 | 0.180 | 0.343 | 0.710 | 0.160 | -0.276 | 0.711 | 0.236 | 0.317 | 0.683 | 0.286 | -0.340 |
| Uv        | -0.887 | -0.330 | -0.265 | -0.898 | -0.341 | 0.240 | -0.852 | -0.413 | -0.252 | -0.858 | -0.401 | 0.248 |
| VRio-i    | 0.960 | -0.184 | 0.106 | 0.961 | -0.183 | -0.104 | 0.972 | -0.099 | 0.101 | 0.965 | -0.100 | -0.119 |

Figure 5. Scree plot to find the factorial loadings.
Fixed nonlinear regression models

The general purpose of multiple regression is to learn more about the relationship between several independent variables and a dependent variable. In multiple regression, the regression coefficient \( R \) can assume a value between 0 and 1. This study employs the following set of standard functions (including: \( X^2, X^3, X^4, X^5, \sqrt{X}, \ln X, \log X \), and \( 1/X \)) to specify nonlinear transformations. The above determined physical and chemical characteristics of the coal samples, including the proximate, ultimate analysis, and petrographic analyses data sets, were considered as the independent parameters and the susceptibility indices CPT, CPT\(_u\), U\(_v\), and VR\(_{io-i}\) were taken as dependent parameters, where the dependence of these variables were considered sequentially. The fitness of each model equation is determined by an analysis of the computed regression coefficients, level of significance, and standard error. Accordingly, 12 model equations were tested for each of the susceptibility indices. A summary of the fitness of each of these models to each of the susceptibility indices in terms of the computed R-Squared, adjusted R-square, and standard error of mean are presented on Table 9. “R-Squared” value of these model equations obtained from nonlinear

![Figure 6. Projection of variables on the factor plane.](image)
regression analysis ranges from 0.319 to 0.955, and the standard error mean (SEM) ranges from 0.084 to 9.329. To develop each model equation, it has been observed that model was significant in the range 0.0 to 0.211. Figure 8 (a) to (l) present a comparative nonlinear plot of the predicted and observed values for the four susceptibility indices. It may be observed from Table 9 that the model equation developed for the four different susceptible indices gives the highest level of significance and fixed nonlinear regression analysis indicates maximum "R-Squared" value of 0.6855 with proximate analysis. Therefore, by using multiple fixed nonlinear regression analysis of the experimental data it may be concluded that the susceptibility index, i.e., unaltered vitrinite (U_v), may be used to categorize/classify the coal seam. It also correlates with other standard methods like CPT and CPT_u. Similarly, the results suggest that the VR_{io-i} method should be explored further by employing a larger number of coals to assess the propensity spontaneous combustion of coal.

**Conclusions**

The study has presented the results of a series of analytical investigations to characterize the characteristic properties (proximate, ultimate, and GCV), petrography
Table 9. Measure of fit of experimental data to fixed multiple nonlinear regression models.

| Sl. No. | Equation | R² | Adjusted R² | P level | Standard error of estimate |
|--------|----------|----|-------------|---------|--------------------------|
| 1      | Proximate Analysis (M, A, VMdaf) |
|        | CPT = 110.286 + 2.092 + 14.486√A | 0.7575 | 0.6535 | 0.0147 | 6.9908 |
|        | CPTu = 115.079 – 1.175 + 84.854 log A | 0.7283 | 0.6119 | 0.0215 | 6.2819 |
|        | Uv = –137.33 + 2.725 – 60.722√A + 6270.618 | 0.7558 | 0.6904 | 0.0100 | 6.6088 |
|        | VRio-1 = –1.791 – 0.180M + 0.002A + 0.072VMdaf | 0.6855 | 0.5507 | 0.0352 | 0.1102 |
| 2      | Ultimate Analysis and GCV (Cdaf, Odaf, GCV) |
|        | CPT = –540.402 + 8.506Cdaf + 5.523Odaf – 0.077GCV | 0.7832 | 0.6904 | 0.0100 | 6.6088 |
|        | CPTu = 551.385 – 0.033Cdaf² + 690.337 – 6.34GCV | 0.6369 | 0.4814 | 0.0567 | 7.2620 |
|        | Uv = –3285.07 + 36.43Cdaf + 29.86Odaf | 0.6965 | 0.5664 | 0.0313 | 9.3291 |
|        | VRio-1 = 4.004 – 0.049Cdaf – 0.017Odaf | 0.4539 | 0.2199 | 0.2117 | 0.1453 |
| 3      | Vitrinite Reflectance |
|        | CPT = 111.518 + 61.355VRi | 0.6244 | 0.5305 | 0.0199 | 8.1381 |
|        | CPTu = 190.611 + 35.359VRi | 0.3194 | 0.1493 | 0.2144 | 9.3007 |
|        | Uv = 15.927 + 40.154VRi | 0.8412 | 0.8016 | 0.0006 | 6.3107 |
|        | VRrio-1 = 0.107 – 0.458VRi | 0.7913 | 0.7391 | 0.0019 | 0.0840 |

Subsequent cross correlation studies suggest potential relationships between coal characteristics properties with the changes in morphology observed in laboratory heat treated coal samples and the liability of such coals to spontaneously combust. This new method requires minimal equipment or preparation of the coal samples. A TGA treated coal samples and the liability of such coals to spontaneously combust. This new method could even potentially be used to generate these samples individually for block preparation and petrographic analysis. Total 11 coal samples examined in this study samples, 1, 2, 3, and 4 exhibit properties that confirm the susceptibility of the coals to spontaneous combustion and this agrees with known behavior from the mine sites themselves. The one exception was in the analyses of sample number 5 (Table 1) collected from the Enna surface coal mine, where local geotechnical conditions and the presence of old near surface workings significantly influence the onset of spontaneous heating. It was also concluded that coal samples collected from the same coal seam but at different depths behave similarly under laboratory conditions and the liability of a coal to spontaneous combustion is not dependent upon the depth of the coal sample within the coal seam. The 11 coal samples studied were divided into three categories as per their susceptibility to spontaneous combustion, i.e., low (first cluster: coal samples 1, 2, and 4), medium (second and third cluster: coal samples 3, 7, 8, 9, and 10) and high (fourth cluster: coal samples 5, 6, and 11).

It is concluded that the proposed morphology study (Urio) delivers an alternative measure of the liability of a coal to spontaneous combustion as compared to two crossing point temperature methods (including CPT and CPTuio). A positive change in reflectance (Vrio-1) of a coal sample pre- and postheating in the muffle furnace described above, recommends that further studies be performed to classify the susceptibility of the coal to spontaneous combustion.

A chemometric analysis of the intrinsic properties of the coal samples, i.e., moisture, ash, volatile matter, carbon, and oxygen on daf basis confirms that these parameters exhibit a positive correlation to the spontaneous combustion susceptibility indices whereas
hydrogen, fixed carbon, vitrinite, inertinite, and liptinite have no correlation with susceptibility indices. Principal component and classification analysis concludes that $U_v$ gives a better indicator for study of spontaneous combustion of coal as compared to CPT and CPT$_u$ which further corroborates the experiments. Fixed multiple nonlinear regression analysis verifies the same.

Figure 8. Fixed multiple nonlinear comparative plot of actual to predicted data.
Acknowledgment

The authors are obliged to Ministry of Human Resources and Development, Government of India, and Council of Scientific and Industrial Research (CSIR) for their kind permission to avail the above fellowship. Authors acknowledge thanks to all staffs of Mine Fire, Ventilation, and Miner’s Safety Research Group, CSIR-CIMFR for necessary help for sample collection.

Figure 8. (Continued.)
**Funding**

This work was supported by the Commonwealth Scholarship Commission [INCS-2010-192].

**References**

Arisoy, A., and B. Beamish. 2015. Mutual effects of pyrite and moisture on coal self-heating rates and reaction rate data for pyrite oxidation. *Fuel* 139:107–14. doi:10.1016/j.fuel.2014.08.036.

Avila, C., T. Wu, and E. Lester. 2014. Petrographic characterization of coals as a tool to detect spontaneous combustion potential. *Fuel* 125:173–82. doi:10.1016/j.fuel.2014.01.042.

Avila, C. R. 2012. Predicting self-oxidation of coals and coal/biomass blends using thermal and optical methods. PhD thesis, University of Nottingham.

Banerjee, S. C. 2000. *Prevention and combating mine fires*, 33. Oxford and IBH Publishing Co. Pvt. Ltd., J. P. House, Delhi.

Benedict, L. G., and W. E. Berry. 1964. Recognition and measurement of coal oxidation. Bituminous Coal Research Report, Monroeville, PA. p. 41.

Benfell, K. E., B. B. Beamish, and K. A. Rodgers. 1997. Effect of resinite on the combustion of New Zealand subbituminous coal. *Thermochimica Acta* 298 (1–2):119–22. doi:10.1016/S0040-6031(97)00142-1.

BS-6127-5. 1995. *Petrographic analysis of bituminous coal and anthracite – Part 5: Method of determining microscopically the reflectance of vitrinite*. London, UK: British standards institute.

BS-ISO-7404-3. 2009. *Methods for the petrographic analysis of coals – method of determining maceral group composition*. British standards institute, London, UK.

Calemma, V., G. Del Piero, R. Rausa, and E. Girardi. 1995. Changes in optical properties of coals during air oxidation at moderate temperature. *Fuel* 74 (3):383–88. doi:10.1016/0016-2361(95)93471-O.

Chandra, D. 1958. Reflectance of oxidized coals. *Economic Geology* 53 (1):102–08. doi:10.2113/gsecongeo.53.1.102.

Chandra, D. 1962. Reflectance and microstructure of weathered coals. *Fuel* 41:185–93.

Chandra, D. 1965. Reflectance of Indian Coals. *Quarterly Journal of Geolgical Mining and Metallurgy Society, India* 37 (1):37

Chandra, D. 1975. Fundamentals of coal petrology - oxidized coal. In *Stach's textbook of coal petrology*, eds. E. Stach, M. T. H. Mackowsky, M. Teichmüller, R. Teichmüller, G. H. Taylor, and D. Chandra, 159–64. Berlin-Stuttgart.

Chandra, D., P. Behera, N. C. Karmakar, and M. N. Tarafdar. 1991. An appraisal of spontaneous combustion of Ib: Valley coals of Orissa. *Minetech* 12 (3):39–44.

Chandra, D., and Y. V. S. Prasad. 1990. Effect of coalification on spontaneous combustion of coals. *International Journal of Coal Geology* 16 (1–3):225–29. doi:10.1016/0166-5162(90)90047-3.

Chen-Brauchler, D., U. Meyer, S. Schlömer, J. Kus, V. Gundelach, M. Wuttke, C. Fischer, and H. Rueter. 2009. *Estimation of near subsurface coal fire gas emissions based on geophysical investigations*. American Geophysical Union, Fall Meeting 2009, San Francisco, California, USA.

Cloke, M., and E. Lester. 1994. Characterization of coals for combustion using petrographic analysis: A review. *Fuel* 73 (3):315–20. doi:10.1016/0016-2361(94)90081-7.

Cloke, M., E. Lester, M. Allen, and N. J. Miles. 1995. Automated maceral analysis using fluorescence microscopy and image analysis. *Fuel* 74 (5):659–69. doi:10.1016/0016-2361(94)00014-1.

Crelling, J. C., R. H. Schader, and L. G. Benedict. 1979. Effects of weathered coal on coking properties and coke quality. *Fuel* 58:542–46. doi:10.1016/0016-2361(79)90175-3.

Deng, J., X. Ma, Y. Zhang, Y. Li, and W. Zhu. 2015. Effects of pyrite on the spontaneous combustion of coal. *International Journal of Coal Science & Technology* 2 (4):306–11. doi:10.1007/s40789-015-0085-y.

DGMS Cir.Tech.3/1975. 1975. *Determination of crossing point and ignition point temperature of coal seams for assessing their proneness to spontaneous heating*. Dhanbad, India: DGMS.

Gray, R. J., A. H. Rhoades, and D. T. King. 1976. Detection of oxidized coal and the effect of oxidation on the technological properties. *Trans Soc Min Eng AIME* 260 (4):334–41.
Ingram, G. R., and J. D. Rimstidt. 1984. Natural weathering of coal. Fuel 63:292–96. doi:10.1016/0016-2361(84)90002-4.

IS-436-Part-1/Sec-1. 1964. Methods for sampling of coal and coke: Part 1 Sampling of coal, Sec 1 Manual sampling (first revision) (Amendment 1). Bureau of Indian Standards. New Delhi. 1: 3–23.

Ivanova, A. V., and L. B. Zaitseva. 2006. Influence of oxidability of carboniferous coals from the Dobrudja foredeep on vitrinite reflectance. Lithology and Mineral Resources 41 (5):435–39. doi:10.1134/S002449020605004X.

Kruszewska, K. J., and V. M. Du Cann. 1996. Detection of the incipient oxidation of coal by petrographic techniques. Fuel 75 (6):769–74. doi:10.1016/0166-2361(95)00264-2.

Kruszewska, K. J., B. C. J. Labuschagne, and V. M. Du Cann. 1996. Relating coal oxidation and hydrophobicity: A petrographic approach. Fuel 75 (14):1611–16. doi:10.1016/S0016-2361(96)00154-8.

Kus, J., and M. Misz-Kennan. 2017. ICCP, Coal weathering and laboratory (artificial) coal oxidation. International Journal of Coal Geology 171:12–36. doi:10.1016/j.coal.2016.11.016.

Lester, E., M. Allen, M. Cloke, and N. J. Miles. 1994. An automated image analysis system for major maceral group analysis in coals. Fuel 73 (11):1729–34. doi:10.1016/0166-2361(94)90160-0.

Lowenhaupt, D. E., and R. J. Gray. 1980. The alkali-extraction test as a reliable method of detecting oxidized metallurgical coal. International Journal of Coal Geology 1 (1):63–73. doi:10.1016/0166-5162(80)90006-3.

Marchioni, D. L. 1983. The detection of weathering in coal by petrographic, rheologic and chemical methods. International Journal of Coal Geology 2 (3):231–59. doi:10.1016/0166-5162(83)90002-2.

Misra, B. K., and B. D. Singh. 1994. Susceptibility to spontaneous combustion of Indian coals and lignites. An organic petrography autopsy. International Journal of Coal Geology 25:265–86. doi:10.1016/0166-5162(94)90001-9.

Misz-Kennan, M., M. Fabiańska, and S. Ćmiel. 2007. Organic components in thermally altered coal waste: Preliminary petrographic and geochemical investigations. International Journal of Coal Geology 71 (4):405–24. doi:10.1016/j.coal.2006.08.009.

Misz-Kennan, M., and M. Fabianska. 2010. Thermal transformation of organic matter in coal waste from Rymer Cones (Upper Silesian Coal Basin, Poland). International Journal of Coal Geology 81 (4):343–58. doi:10.1016/j.coal.2009.08.009.

Misz-Kennan, M., and M. J. Fabiańska. 2011. Application of organic petrology and geochemistry to coal waste studies. International Journal of Coal Geology 88 (1):1–23. doi:10.1016/j.coal.2011.07.001.

Mohalik, N. K. 2013. A study of spontaneous heating of Indian coals. PhD Thesis, Faculty of Engineering, University of Nottingham, UK

Morris, R., and T. Atkinson. 1988. Seam factor and the spontaneous heating of coal. Mining Science and Technology 7 (2):149–59. doi:10.1016/S0167-9031(88)90538-5.

Nandi, B. N., L. A. Ciavaglia, and D. S. Montgomery. 1977. The variation of the microhardness and reflectance of coal under conditions of oxidation simulating weathering. Journal of Microscopy 109 (1):93–103. doi:10.1111/jmi.1977.109.issue-1.

Ndaji, F. E., and K. M. Thomas. 1995. The effect of oxidation on the macromolecular structure of coals. Fuel 74:932–37. doi:10.1016/0016-2361(95)00019-2.

Nimaje, D., and D. Tripathy. 2016. Characterization of some Indian coals to assess their liability to spontaneous combustion. Fuel 163:139–47. doi:10.1016/j.fuel.2015.09.041.

Ogunsola, O. I., and R. J. Mikula. 1990. A study of spontaneous combustion characteristics of Nigerian coals. Fuel 70:258–61. doi:10.1016/0016-2361(91)90162-4.

Pattanaik, D., P. Behera, and B. Singh. 2011. Spontaneous combustibility characterisation of the chirimiri coals, Koriya district, Chhatisgarh, India. International Journal of Geosciences 2 (3):336–47. doi:10.4236/ijg.2011.23036.

Pearson, D. E., and S. Creaney. 1981. Reflectance of carbonized vitrinites as a measure of oxidation of a coking coal. Fuel 60 (3):273–75. doi:10.1016/0016-2361(81)90193-9.

Peters, W. C. 1978. Exploration and mining geology, 416–25. New Jersey, USA: John Wiley & Sons.

Misz-Kennan, M., M. Fabiańska, and J. Ciesielczuk. 2015. Chapter 14 - Thermal Transformations of Waste Rock at the Starzykowiec Coal Waste Dump, Poland A2 - Stracher, Glenn B. In Coal and Peat Fires: A Global Perspective, eds. A. Prakash and E. V. Sokol, 387–429. Boston: Elsevier.
Rencher, A. C. 2002. *Methods of Multivariate Analysis*. New Jersey, USA: John Wiley & Sons.

Ribeiro, J., I. Suárez-Ruiz, C. R. Ward, and D. Flores. 2016. Petrography and mineralogy of self-burning coal wastes from anthracite mining in the El Bierzo Coalfield (NW Spain). *International Journal of Coal Geology* 154:92–106. doi:10.1016/j.coal.2015.12.011.

Stach, E., M. T. Mackowsky, M. Teichmüller, G. H. Taylor, D. Chandra, and R. Teichmüller. 1982. *Stach’s textbook of coal petrology*, 535. Berlin, Germany: Gebrüder Borntraeger.

STATISTICA-7.0. 2004. *Electronic manual statistica 7*. San Francisco, USA: Francisco Partners.