Petrography and geochemistry of exported Colombian coals: Implications from correlation and regression analyses

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ABSTRACT
The petrology of coal, as determined by the microscopic analysis of organic and inorganic elemental constituents of coal, provides information on its nature and characteristics. Coal is used worldwide in the production of thermal energy and coke. In the case of high-quality Colombian coals, only limited studies have been carried out. The present study presents the geochemical, mineralogical, and petrological characteristics of coal samples collected from Puerto Drummond in Cienaga, Colombia, to predict their potential uses. Therefore, the ultimate, proximate, petrographic, gross caloric value (GCV), ash fusion temperature, Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), X-ray fluorescence (XRF), and Thermogravimetric (TG) analyses were employed in this study. The petrological analysis reveals that Colombian coals are naturally more abundant in vitrinite (72%) than inertinite (14–23%) macerals. Silicates and aluminosilicates are the main minerals present as fine particles mixed with macerals. The XRD and FTIR analyses show that the most common and dominant minerals are quartz, while gypsum, hematite, calcite and mica occur in lesser amounts. The oxide composition of the coal ashes consists of 51–58% SiO₂ and 18–25% Al₂O₃ as determined by XRF analysis. In correlation and regression analysis, the moisture content shows a reverse correlation with GCV (Regression value of R² = 0.68). This study helps researchers to comprehend the importance of Colombian coals and presents various techniques for characterisation of coals.

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1. Introduction

Colombia has coal reserves of varying quality distributed across the country. The discovered samples reportedly have a high potential to fulfil the public demand for many hundred years (Lopez & Ward, 2008). Coal is considered as a significant source of energy, and it is one of the most essential resources in South and Central America, which ranks the second with its proved reserve of 4.881 Gt (in anthracite and bituminous coal) in the world. The most significant annual coal production in the region is approximately 62.5 Mt, approximately 1.7% of the world’s total. The growth of coal production is close to 5% (British Petroleum Company (BP), 2017). The International Energy Agency (IEA) reported that Colombia is the fourth largest coal exporter in the world (IEA, 2017), with most of the coal sourced from the Caribbean Region. The Cesar Department, with a population of 1 million and an area of 22.9 km² in the Caribbean Region, is one of the most critical departments in terms of coal production, taking up to 43% of the country’s total coal production, and leading the country to consider it as the most significant coal producer.
Loma. The first is located in the municipality of the same name and is subdivided into two zones: La Jagua and Cerro Largo. The two largest companies that mine these areas are Drummond and Glencore-Prodeco. Their production accounts for nearly 95% of the total production in the department. These companies manage mining and transportation of coal directly from mines to ports located in Cienaga (Magdalena Department) and load vessels for export to Europe, Asia, North, Central and South America (Silva et al., 2012; Drummond, 2017). The lack of fundamental data for these exported coal deposits has influenced many aspects of utilization and safety related to coal in the country.

Coal petrography is the microscopic and macroscopic representation of coal materials, i.e., the elemental composition, mineral matter; maceral groups, and vitrinite reflectance, which is considered as a rank indicator. There are three macerals: vitrinite, liptinite, and inertinite (Hower et al., 1998; Ao et al., 2012). The petrology of organic and inorganic coals along with coal rank determines coal quality (Hower et al., 1998; Ao et al., 2012). So far, there have been only limited studies on the petrology and geochemistry of coal samples from Puerto Drummond in Cienaga, Colombia.

Therefore, the main objective of the present study is to characterize and evaluate the physico-chemical, petrographic and calorific characteristics of exported Colombian coal through various techniques including proximate, ultimate, ash fusibility temperature, Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD) spectroscopy, X-ray Fluorescence (XRF) spectroscopy and Thermogravimetric analyses (TGA). The intercorrelation between the physico-chemical characteristics and macerals with the gross calorific value (GCV) of the coals is graphically examined through regression analysis to establish the relationships between these variables and potential applications.

2. Materials and methods

2.1. Sampling

Standard (0 × 50 mm) thermal coal samples (19 samples) were collected at Puerto Drummond in Cienaga, Colombia (Fig. 1A & B). Samples were prepared using a 5-kg representative split screened through a No. 4-mesh sieve and then divided for each analysis (International Organization for Standardization, 2006).

2.2. Analytical procedures

2.2.1. Chemical analysis

The determination of total moisture of coal samples was carried out in two stages. In the first stage, each sample was crushed to a top size of No. 8 mesh, 2.36 mm, and dried in a drying cabinet (Preiser Scientific Stainless Steel Desiccating Cabinet) at 40 °C. In the second stage, the sample was dried in a drying furnace (Thermo Scientific™ Thermolyne Industrial Benchtop Muffle Furnace) at 108 °C for 3 h (International Organization for Standardization, 2008b, International Organization for Standardization, 2010c).

For proximate analysis, each sample was pulverised to a top size of No. 4 mesh, 180 μm, to determine analysis moisture, ash and volatile matter of samples following ISO standard methods (ISO1171:2010; ISO11722: 2013). The moisture content for the determination was carried out in a Carbolite Model SNF furnace. A coal sample was heated at a temperature of 820 °C with a rate of 15 °C/min for 90 min after which samples were burned for 90 min more up to 815 °C with a rate of 15 °C/min (ISO1171: 2010). For volatile matter (VM) determination, each sample was heated at 900 °C in a muffle furnace (Carbolite AAF 1100, United Kingdom) without contact with air for 7 min and then cooled at ambient temperature for 7 min and in a desiccator (ISO562: 2010) for another 7 min. The determination of carbon, hydrogen, and sulphur contents was performed using an ELTRA CHS 580 analyzer (ISO29541: 2010; International Organization for Standardization, 2006b). This consisted of combustion of 250 mg of sample at 1350 °C in an oxygen flow. The CHN contents were then determined using IR spectrometry. The products of combustion are mainly carbon dioxide, water vapour, and sulphur oxides (International Organization for Standardization, 2005).

The determination of the GCV was carried out in a PARR 6200 Isoperibol Calorimeter based on the bomb calorimetric method. Hence, the net calorific value was determined using pure oxygen, water, and benzoic acid as standard and calculated from the heat capacity of the system (ISO 1928: 2009).

The ash fusibility temperature was carried out in a muffle furnace (Carbolite) using a test piece made of coal ash sample heated at 1000 °C and a rate of 10 °C/min up to 1500 °C. The temperatures at which characteristic changes of the form occur were recorded (ISO540: 2008). The crucible swelling number determination was carried out in a Carbolite Model SNF furnace. A coal sample was heated at a temperature of 820 °C in a covered crucible under standard conditions. The obtained coke button was classified in comparison with the standard profile schemes (ISO 501: 2012). Hardgrove grindability index was determined using a Hardgrove grinding machine (Preiser Scientific) at 60 revolutions and a Ro-Tap sieve shaker (W.S. Tyler). The milling index was determined from the granulometric analysis of the milled product referenced to standard test materials in the literature (International Organization for Standardization, 1994).

2.2.2. Organic petrographic analysis

For each coal sample, epoxy-bound particulate pellets (finer than 20-mesh) were prepared to final 0.05 μm polish. Microscopic examination was subsequently carried out on a Leitz Orthoplan microscope at the Center for Applied Energy Research, University of Kentucky, using reflected-light optics and a 50× oil-immersion objective. The maceral identifications of the coal samples were based on the nomenclature of ICCP (ICCP, 1998; ICCP, 2001; Choudhury et al., 2017; Pickel et al., 2017). The mean maximum vitrinite reflectance was measured from 50 points per coal, using polarised light and a 546-nm filter.

2.2.3. Thermal analysis

Thermogravimetric analysis (TG-DTG) was carried out under non-isothermal conditions using the Thermal Analyzer (Model: TGA 701, LECO). For each test, about 25 mg of sample was placed in an alumina crucible and heated from room temperature (RT) to 900 °C based on the non-isothermal heating rate of 10 °C/min in an oxygen environment.

2.2.4. Fourier transform infrared spectroscopy

FTIR analysis was carried out in a PerkinElmer System 2000 apparatus in the range of 4000 cm⁻¹ to 400 cm⁻¹. Samples were thoroughly mixed with the Potassium Bromide (KBr) powder in an agate mortar. This was followed by pelletization of the mixture. The FTIR spectra of the coals were subsequently analysed using the software spectrum V-3.02 (Version 3.02.01, PerkinElmer) in advance mode (Choudhury et al., 2017).
2.2.5. X-ray diffraction analysis

X-Ray diffraction (XRD) was performed using the X-Ray diffractometer (Rigako, Ultima IV). The resulting X-ray diffraction data of the coal samples were analysed with starting angle set at 2.00° and stopping angle set at 90.00° and step angle 0.02° with a scanning rate of 1° C/min and target Cu–Kα (λ = 1.7902Å). The library database ‘Rigaku PDXL 1.2.0.1’ programme was used for the identification of peaks (Saikia et al., 2014).

2.2.6. X-ray fluorescence spectroscopic analysis

The determination of minor and major elements in coal samples was carried out by X-ray fluorescence (XRF) test method specifications (Saikia et al., 2015a,b). Ashed samples were mixed with cellulose as a binder in a weight ratio of 3:0.6 g and then pellets were made by using a VANEOX -25 t manual press. The sample pellets were then analysed using an Epsilon 3XL benchtop spectrometer with Ag anode X-ray tube and 50 μm thin beryllium window for high sensitivity for light elements (Na, Mg, Al, and Si).

3. Results and discussion

3.1. Chemical characteristic of coal

The moisture and ash content of each coal sample expressed on dry basis is shown in Table 1. The total moisture ranges from 6.05 to 16.09%, whereas the ash was in the range from 6.26 to 8.44%. However, other studies observed that the total moisture content of the Venezuelan samples ranges from 1 to 6 wt% (Hackleya et al., 2005). In these studies, total sulphur content was low with a range of 0.57–0.90%, which indicates this type of coal is suitable for energy generation with restrict regulation of sulphur emission. Low-sulphur coals are generally formed in a fluvial environment, while coals with high sulphur content are deposited in the fresh water alkaline environment affected by seawater incursion with effects of differing groundwater conditions, post-depositional, and authigenic mineralization (Kostova, and Isaeva, 2016).

There are two coals with higher calorific value, sample 1440 with 31.15 MJ/kg and sample 1448 with 31.42 MJ/kg, which can be related to their higher volatile matter content, 40.73%, and 40.93%, respectively. The latter is corroborated with samples 1471 and 1472 whose calorific value and volatile matter content are directly proportional.

3.2. Petrographic characteristics of coal

The petrographic data for the investigated coal samples are given in Table 2 while the photomicrographs of representative coal specimens that showed some vital mineral components are depicted in Fig. 2. The coals are characteristically richer in vitrinite than in inertinite macerals with over 70% vitrinite mainly occurring in the form of collodetrinite. Furthermore, about 14–23% inertinite is present mainly in the form of fusinite and semifusinite. However, the samples with high inertinite contents exhibit low proportions of vitrinite and vice-versa. In addition to the macerals, the coal samples also contain 0.5–5.1% of total mineral components. These levels are somewhat low considering the export potential of the coal types, which are beneficiated for this purpose (Khare and Baruah, 2010). Silicates are the main minerals found as fine particles mixed with fragmented macerals (Fig. 2-D) and are attributed to detrital origin (Vassilev and Vassileva, 1996). The Hard grove index (HGI) of most of the coal samples characterize the grinding of the coals and indicator of mill performance. The coals are moderately hard and depend on the vitrinite characteristics as observed above.

3.3. Thermogravimetric properties of coals

The TG-DTG data (Fig. 3) of the coal samples mainly indicate three temperature regions. The DTG curve of all coal samples shows the same trend in temperature regions in having three peaks due to
the presence of similar chemical bonds in their molecular structures. The first region (Fig. 3.1) occurs between 33-195 °C which indicates the release of moisture content from the coal samples (Huang et al., 1996). The maximum loss of water is observed at 89 °C. The second region occurs between 195 °C and 276 °C, where maximum mass loss occurs due to primary devolatilization and char oxidation (Huang et al., 1996). In the second region of the TGA, the graph shows a lower peak temperature due to the reduction of H-bonding occurs (Sharma et al., 2016).

Furthermore, a double peak can be observed in the DTG curves of all coal samples, which could be attributed to the stepwise release of volatile matter. In the third region, observed from 313 °C to 851 °C, the coals begin to oxidise due to the release of CO, CO2, and CH4 from volatile matter (Salkia et al., 2014). In this region, the decomposition of mineral matter (quartz, kaolinite etc.) occurs in coal (Sharma et al., 2016) upon combustion, which starts to decompose in the temperature range of 300–350 °C up to the final temperature of 850–900 °C. The thermal decomposition of coals
occurs at a high temperature which leads to the formation of hydrocarbons and tar through the breaking of bonds (Baruah and Khare, 2007; Khare et al., 2011). The maximum loss of volatile matter is observed at 185°C. These analyses indicate the usefulness of Colombian coals in hard coke making and other industrial purposes (Saikia et al., 2009).

### 3.4. Functional group analysis

The FTIR spectrum (Fig. 4) of the coal samples reveals sharp peaks in the region of 3000-3600 cm⁻¹ due to the stretching vibration of aromatic O–H (phenolic group) and amines (N–H). In this region, the stretching band also indicates the presence of quartz due to the hydroxyl linkage in the clay mineral present in coal (Saikia et al., 2015a,b). The peaks in the 2800-3000 cm⁻¹ range indicates the presence of alkane group (C–H). The absorption peak at 2300-2326 cm⁻¹ is observed due to the presence of the silane group (S–H) of the coal samples. The strong absorbance peak occurs at 1506 ± 60 due to stretching of the aromatic C=C ring. Deformation bands (C–H def) occur in the range between 1400-1450 cm⁻¹ and the stretching frequency in the range between 1000-1100 cm⁻¹ due to the presence of aliphatic ethers and alcohols in various intensity (Khare and Baruah, 2010). This region (1030 ± 30) also indicates the presence of sulfoxide (S=O) and probably some kaolinite minerals in the coal samples (Painter et al., 1981). The absorption peak at 700–900 and 800-1000 cm⁻¹ are observed due to the formation of kaolinite, hematite, and illite (Wilson, 1994; Saikia et al., 2015a,b). The stretching bands at 530, 541, and 691 cm⁻¹ occur due to S–S bonds of sulfides and the presence of primary and secondary thiol (Khare and Baruah, 2010; Saikia et al., 2015a,b). The peaks at 468 and 472 are assigned to the stretching vibration of sulphonamide (S=N). The absorption peak at 650 ± 10 reveals the presence of chlorite and the peak at 472 indicates the presence of montmorillonite (Speight, 1983). The clay particles act as catalyst during the cracking of volatile matter which cannot be adsorbed on coal on cooling (Saxby et al., 1992).

### 3.5. XRD-mineralogy

The presence of minerals in the coal samples and their qualitative assessment were carried out by X-ray diffraction. The minerals are identified in the coal samples based on the d-spacing values of the XRD peaks (Dutta et al., 2017) (Table 3). The coal samples show the presence of quartz, gypsum, hematite, calcite, and mica, as presented in Fig. 5. Quartz is the standard and dominant mineral present in all the samples except for sample 105 and sample 1446. A prominent peak of quartz is observed in most of the samples due to its high stability (Dutta et al., 2017). Similarly, quartz and hematite were also present in the FTIR analysis of coal samples. The detailed assignments of theta and d-values for the detection of minerals are shown in Table 3.

### 3.6. Coal ash analysis

The chemical composition (%) of the coal ashes were determined by XRF analysis as presented in Table 4. The results indicate that the coal ash is largely composed of 51–58% of SiO₂ and 18–25% of Al₂O₃. Furthermore, the ash also contains low percentages of Mn₃O₄, P₂O₅, SrO, and BaO. The presence of 9–12% of Fe₂O₃ along with pyrite and jarosite occurs in the coals from the northern region.
Colombian mines (López and Ward, 2008). The proportion of CaO in the coal ashes varies from 2 to 4.7% which may be due to the presence of organic matter bound in the clay minerals. The proportion of sulphur as SO$_3$ retained in the ash varies over 1–5.7%. This is attributed to variation in the proportion of pyrite in the feedstock coals.

3.7. Correlation and regression analysis

The relationship between the macerals (or chemical characteristics) with the gross caloriﬁc value (GCV) of the coals was examined by numerical analysis. The statistical variance was computed to establish the numerical relationships between each chemical constituent of the coal samples. Consequently, the interrelationships between the maceral/chemical properties (comprised of the volatile matter, moisture, fixed carbon, ash, vitrinite, inertinite, mineral matter, and ash) and the GCV of coal samples was examined and plotted as presented in Fig. 6.

As observed, the relationship between volatile matter on dry basis and GCV is characterized by an $R^2$ value of 0.4942, which indicates a good measure of maturity or high coal rank. However, the moisture contents show a reverse correlation with GCV ($R^2 = 0.6754$). In comparison, the analysis of US coal samples revealed negative effect or weakly correlation between the moisture content and GCV. This parameter evidently indicates slow ranks of the coal due to the diluents related to GCV (Mesroghli et al., 2009). In this study, the bivariate plots show there is no relationship with r squared values all below 0.2.

4. Conclusions

The study presented an overview of the geochemical, mineralogical, and petrographic characteristics of Colombian coals. The chemical and petrographic characteristics of coal can be used as a first-rate indicator of their potential for combustion, liquefaction, and coke production. From these investigations, the Colombian

Fig. 3. TG-DTG spectra of coal samples showing the thermal properties of the coals [(a)-105, (b)-1409, (c)-1425, (d)-1427, (e)-1438, (f)-1440, (g)-1442, (h)-1446, (i)-1448, (j)-1449, (k)-1465, (l)-1466].

**Table 3**

Assignments of XRD d-values for the detection of mineral phases.

<table>
<thead>
<tr>
<th>Samples</th>
<th>2θ</th>
<th>d-value</th>
<th>Possible minerals</th>
<th>2θ</th>
<th>d-value</th>
<th>Possible minerals</th>
</tr>
</thead>
<tbody>
<tr>
<td>a</td>
<td>20.8</td>
<td>4.958476835 Quartz</td>
<td>l</td>
<td>12.42</td>
<td>8.274718346 Gypsum</td>
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</tr>
<tr>
<td></td>
<td>26.56</td>
<td>3.896651967 Quartz,</td>
<td>m</td>
<td>50.48</td>
<td>2.099149084 Quartz, hematite</td>
<td></td>
</tr>
<tr>
<td>c</td>
<td>12.34</td>
<td>8.328153563 Gypsum</td>
<td>n</td>
<td>12.7</td>
<td>8.093006647 Quartz</td>
<td></td>
</tr>
<tr>
<td></td>
<td>26.62</td>
<td>3.888027156 Quartz,</td>
<td>o</td>
<td>26.56</td>
<td>3.896651967 Quartz</td>
<td></td>
</tr>
<tr>
<td>d</td>
<td>18.46</td>
<td>5.58049251 Quartz</td>
<td>p</td>
<td>26.3</td>
<td>3.934485167 Quartz</td>
<td></td>
</tr>
<tr>
<td>e</td>
<td>26.82</td>
<td>3.85955911 Quartz</td>
<td>q</td>
<td>12.38</td>
<td>8.301349505 Gypsum</td>
<td></td>
</tr>
<tr>
<td>f</td>
<td>21.02</td>
<td>4.907154588 Quartz,</td>
<td>r</td>
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<tr>
<td>g</td>
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<td>4.958476835 Quartz</td>
<td>s</td>
<td>26.56</td>
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<td>4.958476835 Quartz, hematite</td>
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<td>3.85955911 Quartz,</td>
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<td>3.85955911 Quartz</td>
<td>w</td>
<td>68.28</td>
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<td>8.328153653 Gypsum</td>
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**Fig. 4.** FTIR spectra of coal samples showing presence of functional groups (a)-105, (b)-1409, (c)-1425, (d)-1427, (e)-1438, (f)-1440, (g)-1442, (h)-1446, (i)-1448, (j)-1449, (k)-1465, (l)-1466, (m)-1467, (n)-1472, (o)-1493, (p)-1508, (q)-1506, (r)-1510, (s)-3229644.
Fig. 5. XRD analysis of coal samples showing the mineral phases in the coals [(a)-1427, (b)-1446, (c)-1448, (d)-1493, (e)-1510, (f)-105, (g)-1438, (h)-1440, (i)-1442, (j)-1508, (k)-1409, (l)-1425, (m)-1449, (n)-1465, (o)-5329644, (p)-1466, (q)-1467, (r)-1472, (s)-1506] [Q-quartz, H- hematite, G-gypsum, C- calcite, M-mica].

Table 4
Major element composition of coal ashes (815 °C) by XRF spectrometry (wt.%).

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>SiO₂ %</th>
<th>Al₂O₃ %</th>
<th>Fe₂O₃ %</th>
<th>TiO₂ %</th>
<th>MnOx %</th>
<th>CaO %</th>
<th>MgO %</th>
<th>Na₂O %</th>
<th>K₂O %</th>
<th>P₂O₅ %</th>
<th>SO₃ %</th>
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<td>52.81</td>
<td>19.56</td>
<td>10.60</td>
<td>0.95</td>
<td>0.07</td>
<td>3.67</td>
<td>1.60</td>
<td>2.63</td>
<td>1.93</td>
<td>0.22</td>
<td>5.51</td>
<td>0.13</td>
<td>0.33</td>
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<td>1440</td>
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<td>1.57</td>
<td>0.09</td>
<td>2.55</td>
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<td>0.45</td>
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<td>11.62</td>
<td>0.97</td>
<td>0.08</td>
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<td>1.64</td>
<td>2.30</td>
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<td>1.03</td>
<td>0.06</td>
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<td>1.46</td>
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<td>0.06</td>
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<td>1.48</td>
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Coals are vitrinite-rich as the samples contain 0.5–5% of visible mineral components. Silicates were found to be major minerals, and exist as fine particles with partial detrital origin. However, the major minerals identified by XRD and FTIR analyses were quartz, gypsum, hematite, calcite, and mica. Based on the results, the coal samples examined are potentially suitable for energy generation, provided there is strong regulation of sulphur due to the presence of low-sulphur content (0.57–0.90%). The intercorrelation between the chemical characteristics and GCV of the coals reveals that higher moisture content adversely affects the GCV of Colombian coals, based on its reverse correlation with GCV ($R^2 = 0.6754$). Likewise, the macerals (vitrinite, inertinite, liptinite and mineral matter) show poor correlation with GCV ($R^2 = 0.0553$; $R^2 = 0.0425$; $R^2 = 0.0021$; $R^2 = 0.0521$). The results of this petrographic analysis will be helpful in further investigation on the relationship between GCV and coal quality. It is envisaged that the findings of the present

**Fig. 6.** Relationship of chemical characteristics of coal and macerals with GCV.
study will motivate further research studies on the characterisation and utilization of Colombian coals in the nation’s scientific community.

Declaration of competing interest

The authors declare that they have no conflict of interest.

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References


