

# Microstructural relation of macerals with mineral matter in coals from Ib valley and Umaria, Son-Mahanadi basin, India

Vivek Mishra<sup>1</sup> · K. N. Singh<sup>2</sup>

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**Abstract** Coal petrology provides significant inputs for the industrial utilisation of coal and for broad understanding the coal formation and diagenesis. The present paper entails the results of the investigations carried out on the selected coal samples, from Ib valley and Umaria coalfield, using scanning electron microscope (SEM) and X-ray diffraction (XRD) to study the surface microstructures and minerals present in them and the relationship of the finely dispersed mineral matter with the organic constituents. This would further help in evaluating the distribution and chemical character of the mineral matter occurring within the maceral types. Ib valley and Umaria coals are typical Indian (Lower Gondwana) non-coking coals and only scanty data is available on SEM study of these coals. Under SEM examination, it manifests that, the mineral matters of these coal occur as deep intergrowth, massive impregnation, superficial mounting, filling and depletion of micropores, mechanical cavity filling and fusinitic cavity filling.

**Keywords** Coal · SEM · XRD · Umaria coal · Ib valley coal · India

## 1 Introduction

Coal is an organic biological rock (Xie 2015), formed from dead plant remains that accumulated to a certain thickness in a basin and was subsequently covered by the sediments through physical, chemical, and biological processes in an appropriate geological environment over a long geological time. Coal, therefore, contains organic matter and mineral matters in variable quantities. About 95 wt% of mineral matters consist usually of shale, kaolin, sulphide and chlorite (Shirazi et al. 1994; Saikia 2009). The heterogeneity in coal has the prime importance in the structural characterisation of coal (Thomas 1986). Its heterogeneity at the macroscopic level is reflected as banded structures as

shown in Fig. 1; most of the plant materials, except in very low rank coal, appears almost structureless in final product, coal (Seyler 1928; Kroeger 1964; Winston 1988, 1989; Pierce et al. 1991; Stanton and Moore 1991; Moore and Ferm 1992; Shearer 1992). At times, there is difficulty in distinguishing the inorganically-bound minerals and elements from the organically-bound ash-forming elements and minerals (Davidson 1990).

The use of scanning electron microscopy (SEM), offers great opportunity for the study of microstructural features of coal and coal products (Singh et al. 1987; Singh 1989; Singh and Singh 1990, 1995). Finkelman and Stanton (1978) suggest SEM as a brilliant tool for determining the maceral content and amounts of elements and minerals in the maceral. The scanning electron microscope can only deliver qualitative data. However, visual assessment of mineral matter and micro structural features may be carried out by evaluating the viewed surface area of the litho type shielded by mineral matter and by observing the number of places where the same micro structural features appear. Nowadays, computer-controlled advanced SEM is also available through which qualitative as well as quantitative

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✉ Vivek Mishra  
vmishranml@gmail.com

<sup>1</sup> CSIR - National Metallurgical Laboratory,  
Jamshedpur 831007, India

<sup>2</sup> School of Studies in Earth Science, Vikram University,  
Ujjain 456010, India



**Fig. 1** A cross-section through a lump of coal showing vitrain, clarain, durain and fusain bands with mineral matter

mineral analysis is carried out and mineral matter-organic matter association, size distribution are also studied (Galbreath et al. 1996; Gupta et al. 1998; Creelman and Ward 1996; Gottlieb et al. 1991; Kalaitzidis and Christanis 2003; Liu et al. 2005; Saikia and Ninomiya 2011; Saikia et al. 2015; Singh et al. 2015a). To quantify the minerals present in the Ib and Umaria coal samples, X-ray powder diffraction (XRD) was carried out. XRD is the most extended tool to investigate the mineralogy of coal and their crystallisation phases and also to understand the exact nature of their structure and the progressive stages of coalification (Zhou et al. 2010; Mishra and Das 2010; Saikia et al. 2014; Singh et al. 2015b; Valentim et al. 2016; Mishra et al. 2016a, b).

The present paper entails an initial examination of the surface morphology of maceral and their relationship with the mineral matter in some coal samples collected from Ib river valley, Mahanadi basin, Odisha and Umaria coalfield, Madhya Pradesh. Some recent contributions on coal deposits of the Mahanadi valley have been made by Singh et al. 2013, Naik et al. 2016 and Mishra et al. 2016a. Nevertheless, the present study carried out under SEM and XRD would help in identifying their potential utilisation.

## 2 Materials and methods

Thirteen fresh Coal samples were collected from different collieries of Umaria and Ib-valley coalfield. The collected samples are from working face as full-seam channel samples from base to top in sub-seam intervals and were classified as per scheme given by Diessel (1965). The individual coal sample were crushed and pulverised for various analysis. The proximate analysis of the coal samples were carried out by standard methods (IS:1350 1984). The elemental analysis (C, H, N, and S) was conducted using Vario EL-III analyser.

To study the micro-constituents of coal (maceral), the coal samples were crushed to  $-18$  mesh size. The moulds were prepared in cold medium using epoxy-resin and

hardener and were subsequently polished for microscopic study. The maceral analysis was performed on a polarised incident-light microscope with an automatic photographic unit (Wild Photo-automat MPS 45) using established ICCP (1963, 1998, 2001) recommendations.

The SEM study is performed on JEOL scanning electron microscope, model Philips 505. SEM samples were prepared by sprinkling powdered coal samples onto a carbon coated metallic holder followed by gold coating.

For X-ray diffraction (XRD) studies, representative coal samples powdered to 300 mesh size. XRD patterns were recorded on a Rigaku (D/Max III VC) instrument in the  $2\theta$  region of  $2^\circ$ – $60^\circ$ . The obtained peaks in the diffractogram have been identified by the peak finding program. The  $2$ -theta ( $2\theta$ ) peaks were converted into corresponding d-spacing and matched with the Joint Committee on Power Diffraction Standards (JCPDS) database.

## 3 Results and discussion

The proximate and ultimate analysis of coal samples were carried out in triplicate and mean values has been reported in Table 1. In general, all coal samples display high volatile matter (22.3%–34.5 wt% in Umaria coals and 24.0%–34.0 wt% in Ib river coals on dry ash free basis) with a moderate ash yield (10.5–20.0 in Umaria coal and 16.0%–24.2 wt% in Ib river coal on dry basis). In these coals, Umaria coals show low in liptinite content (2.66%–4.35 vol% on mineral matter free basis) where as Ib valley coals contain moderate to high amount of liptinite (3.15%–9.85 vol% on mineral matter free basis). The inertinite content, however, is high (28.97%–65.76 vol% on mineral matter free basis) in these coals (Table 1).

Mineral matters occur in coal in different mode of occurrences. There are many different minerals that behave differently. The main minerals are quartz, metakaolinite, mullite, and rutile, while the common fluxing minerals are anhydrite, acid plagioclases, K feldspars, Ca silicates, and hematite (Creelman et al. 2013; Mishra et al. 2016a, b). Figure 2 represents XRD diffractogram of two samples (Umaria and Ib valley coal). It indicates clearly the presence of quartz (Q), kaolinite (K), siderite (S) and hematite (H) as major mineral phases in both samples. The XRD patterns of both coals are found to show almost similar mineral composition. The identification of minor minerals only by XRD in a multi component system like coal is difficult due to the detection limits (normally at about 0.5%–1%) and peak overlapping (Mishra et al. 2016a).

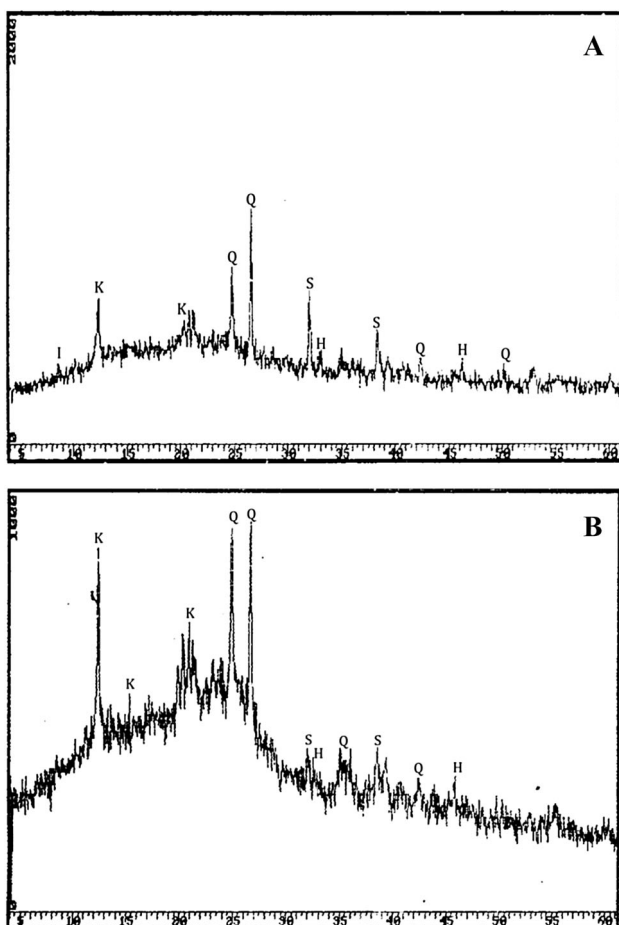
Figures 3 and 4, shows the SEM photographs of the various maceral of the Umaria and Ib valley coals.

Singh et al. (2015a) found a specific micro-structural relation between mineral matter and the coaly substance in

**Table 1** Chemical and petrographic characteristics of Umaria and IB-valley coal

Sample No.	M	VM	A	C <sup>daf</sup>	H <sup>daf</sup>	N <sup>daf</sup>	S <sup>daf</sup>	Vitrinite <sup>mmf</sup>	Liptinite <sup>mmf</sup>	Inertinite <sup>mmf</sup>
UC 1	6.5	30.5	17.8	80.9	5.5	1.00	0.51	53.55	4.35	42.10
UC 2	7.0	32.0	18.2	79.2	5.6	1.01	0.52	55.10	4.04	40.86
UC 3	4.7	22.3	20.0	84.7	5.1	1.04	0.61	41.54	2.66	55.80
UC 4	6.3	34.5	10.5	81.4	5.6	1.06	0.54	56.32	4.23	39.45
IC 1	5.3	31.3	21.0	80.7	5.7	0.79	0.50	58.83	5.80	35.37
IC 2	6.0	29.7	16.0	81.6	5.5	0.77	0.54	65.80	5.23	28.97
IC 3	6.0	27.7	24.2	80.8	5.5	0.79	0.72	49.60	7.45	42.95
IC 4	5.0	34.0	18.5	80.5	5.8	0.75	0.79	33.25	9.85	56.90
IC 5	5.5	26.0	24.0	81.8	5.4	0.87	0.97	57.25	3.15	39.60
IC 6	5.7	27.6	21.7	81.6	5.5	0.74	0.91	31.81	9.32	58.87
IC 7	6.0	24.0	21.0	82.9	5.2	0.72	0.81	33.05	6.95	60.00
IC 8	5.6	30.7	17.3	81.5	5.6	0.74	0.83	28.61	5.63	65.76
IC 9	4.7	29.5	17.2	82.4	5.4	0.74	0.68	36.60	8.40	55.00

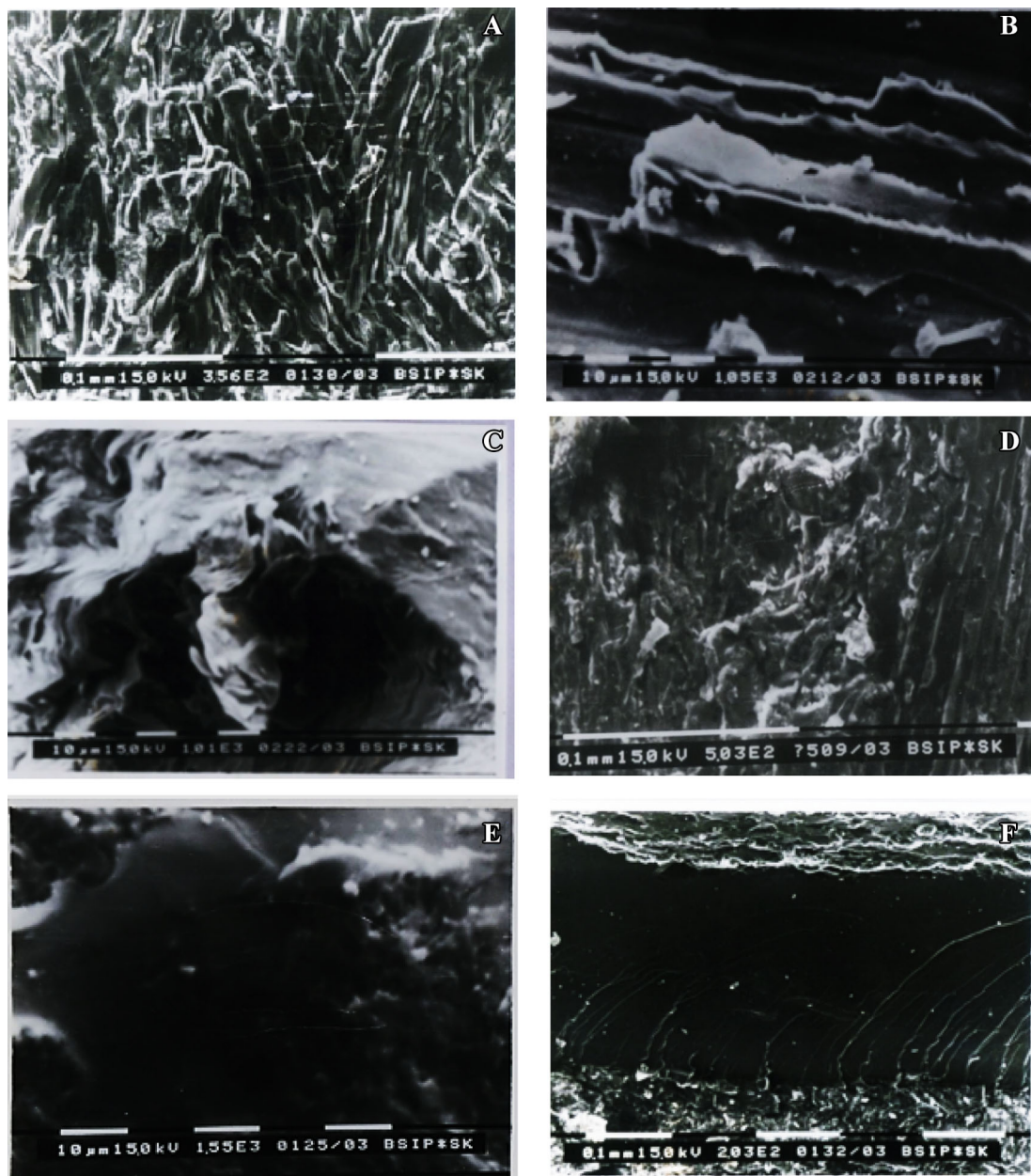
M moisture, VM volatile matter (wt%), A ash yield (wt%), C carbon (wt%), H hydrogen (wt%), N nitrogen (wt%), S sulphur (wt%), *daf* dry ash free basis, *mmf* mineral matter free basis



**Fig. 2** XRD diffractograms of coal samples. **a** Ib-valley coal, **b** Umaria coal. H hematite, I illite, K kaolinite, Q quartz, S siderite

Meghalaya coal. In the moderately dull coal, mineral matter occurs as superficial impregnation and cavity filling; in moderately bright coal as superficial impregnation, cavity filling, and intimate intergrowth; and in bright coals as superficial mounting and pore fillings. In Umaria and Ib valley coal, in general mineral matter is more dominant in dull bands as compared to the brighter ones (Fig. 3d–f). The micro structural relationships between coaly substances and mineral matter in vitrain, appears as amorphous mass with conchoidal fracture or observed mainly superficial mounting (Fig. 3a), superficial blanketing (Fig. 3b, c), deep intergrowth (Fig. 3d) and massive impregnation (Fig. 3c). Saikia (2009) has reported two different morphological types of collotelinite in Assam coals. Those are of typically angular in shape and with parallel laminations. Increase in coal rank appears to increase the number of laminations and give more ordered system of stacked sheets. Particular, in Umaria coal, telinite shows cellular structures with woody matter compressed probably due to pressure (Fig. 3e, f). In case of clarain, characterised by alternate thin bands of vitrain and durain, mineral matter occurs as superficial blanketing and cellular cavity filling. In dull bands it occurs as massive intergrowth and deep impregnations. In durain which is characterised by structureless, compact, residual ground mass, the mineral matter occurs as intergrowth and massive impregnation. This lithotype records the maximum contamination. Finkelman and Stanton (1978) found pyrite concentrated in vitrinite macerals; illite, quartz, and rutile in the carbominerite; and kaolinite in fusinite and semifusinite. Finkelman (1980) has reported sulfides, concentrated in inertinites and vitrinites.





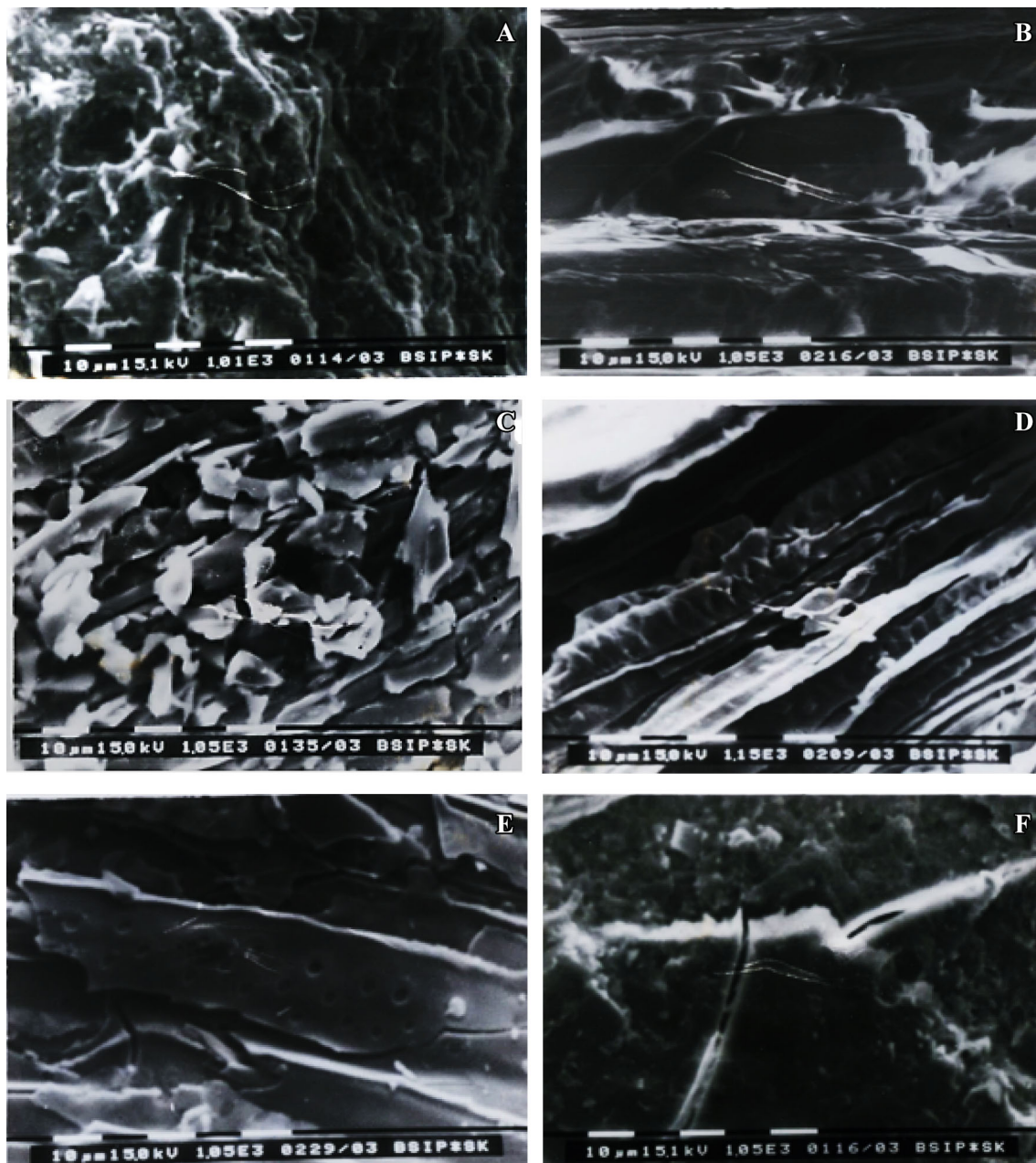
**Fig. 3** SEM photomicrographs a–d showing vitrain band from Umaria and Ib valley with superficial mounting, mineral impregnation, cavity filling and mineral matter filling in tracheids. SEM photomicrographs e, f showing clarain band from the same coalfield with mineral intergrowth, deep impregnation and superficial mounting of mineral matter dominantly of argillaceous siliceous and sideritic composition

Late epigenetic minerals, such as kaolinite, siderite, pyrite, calcite, barite and silica and most commonly the aluminum silicates have been reported by Finkelman (1980) to occur in micro cleats of coal. In the present investigation, the voids are seen to be filled with clayey, sideritic and at places limonitic mineral matter (Fig. 4a, b). Davis et al. (1986) and Saikia (2009) have reported rod-like (needle) cylinders resembling fossilised xylem plant vessels in coal. In case of Ib valley and Umaria coal, generally the concentration of mineral matter is less in fusain. The mineral matter has been found in pitted vessels of parenchyma. The

pitted vessels and mechanical cavities owe their origin to the crushing of cell fibres (Fig. 4c–f).

#### 4 Conclusions

Most of the minerals in the coals of the study area are superficially mounted. Mineral impregnation in cell wall is flocculated with granulated texture. At some places these show relict structures. In cracks and fissures, sideritic mineral matter forms hard ridges and relict structure. Some



**Fig. 4** SEM photomicrographs **a, b** showing durain band from Umari and Ib valley respectively with maximum coating of mineral matter including voids filling with clayey and sideritic mineral matter. SEM photomicrographs **c-f** show fusain band from the same coalfield having minimum interaction with mineral matter. Some crushed cells/vessels are also seen with sporadic mineral inclusion in veins

pits have been distinguished in fusains. These pits are the parts of tracheid and mostly free from any mineral matter specially in Ib valley coals.

The present investigation reveals that durain contains maximum mineral matter contamination while fusain has been minimum. Due to high mineral matter durain appears dull under SEM. In decreasing sequence of mineral matter the lithotype can be arranged as durain > clarain > vitrain > fusain.

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