

Geochemistry of Kasnau-Matasukh lignites, Nagaur Basin, Rajasthan (India)

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Abstract The distribution and verticals variation of geochemical components in the Kasnau-Matasukh lignites of Nagaur Basin, Rajasthan, were investigated using microscopy, proximate and ultimate analyses, Rock–Eval Pyrolysis, X-ray diffraction and Fourier Transform Infrared analyses, and major/minor/trace element determination. The relationship of elements with ash content and with macerals have also been discussed. These lignites are stratified, black, dominantly composed of huminite group macerals with subordinated amounts of liptinite and inertinite groups. They are classified as type-III kerogen and are mainly gas prone in nature. The concentration (in vol%) of mineral matter is seen to increase towards upper part of seam and so is the concentration (in wt%) of the volatile matter, elemental carbon and sulphur. The common minerals present in these lignites are mixed clay layer, chlorite, and quartz as identified by X-ray diffraction study. Compared with world average in brown coal, the bulk concentration of Cu is anomalously high in most of the samples while Cd is 2–3 times high and Zn is high in one band. Based on interrelationship, different pyrite forms are noticed to have different preferential enrichment of various elements. The concentration of disseminated pyrite is more than the other pyrite forms and is followed by discrete pyrite grains and massive pyrite.

Keywords Geochemistry · Macerals · Trace elements · Kasnau-Matasukh lignite · Rajasthan

1 Introduction

Coal is an organo-elastic sedimentary rock composed of lithified plant debris. The inorganic constituents occurring in different forms in coal of all rank are collectively known as ‘mineral matter’. Mineral matter may occur as crystalline solids, dissolved salts in pore waters, organo-metallic compounds, discrete and disseminated grains. The type and quantity of mineral matter in coal depends on nature of vegetal matter, mode of accumulation (allochthonous or autochthonous), tectonic framework of the depositional basin, hydrological conditions, climatic

conditions and the geomorphology of the hinterland. There are different phases during which the mineral matter gets into coals. These include, (i) inherent in organic matter of plants, (ii) syngenetic minerals added during the stage of peat development, and (iii) secondary or epigenetic minerals deposited through circulating waters during the coalification process. Contributions on the significance of mineral matter and trace elements in coal have been made by many researchers. Ward et al. (1999) discussed the trace elements and mineral matter of New South Wales, Australia, in the light of quantitative data obtained through X-ray diffraction study. Mineralogical analysis of these Australian coals was further used for the seam correlation (Ward et al. 2001). A detailed account on the significance of mineral matter in coal is provided by Ward (2002). Li et al. (2010) used electron microprobe study to know the occurrence of non-mineral inorganic elements in macerals of low rank coals while Dai et al. (2013) studied the mineralogical and geochemical anomalies of Late Permian

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coals from southern China and evaluated the influence of hydrothermal fluids and terrigenous materials. They also carried out study on the elements and phosphorus minerals in the Jurassic coals of Tibetan Plateau (Dai et al. 2015a, b). While working on mineral matter in coal, Ren (1996) discussed the significance of trace elements in coal revealing geologic information about coal-bearing sequence formation, depositional condition and regional tectonic history of the basin. Trace elements get accumulated in coal in two ways, through plants and animals, and through geologic processes during peat and post peatification stages (Bouška et al. 2000). The impact of trace elements on environment depends upon their modes of occurrence (mobility), concentration, and toxicity (Finkelman 1995; Dai et al. 2005) and thus, study of trace elements would help in formulating strategies to combat pollution related to coal combustion (Querol et al. 2001; Dai et al. 2005; Wang et al. 2008; Tang et al. 2009). There are certain elements like As, Be, Cd, Cr, Co, Cu, Pb, Mn, Hg, Mo, Ni, Sr, U, V, and Zn which are environmentally more sensitive and have their impact on environment when released into atmosphere especially after their combustion in the thermal power plants (Pickhardt 1989; Turiel et al. 1994; Singh et al. 2011, 2012, 2014; Prachiti et al. 2011; Singh and Singh 2013). The mean abundance of elements in coal is also important for geochemical comparisons (Ketris and Yudovich 2009). Silicates, carbonates and sulphates are the major minerals in coal which consists most of the elements but some elements such as Ge, B, Br, Be, and Cl are associated with the organic matter (Finkelman 1995).

Though, various workers have contributed on the geological aspects of the Kasnau-Matasukh lignites of Nagaur basin, but no work has been carried out on the petrological and geochemical aspects of these lignites. Therefore, the present study has been undertaken to see the distribution and variation of the geochemical constituents, including major/minor/trace elements, vertically along the seam profile of these lignites. Further, the inter-relationships among the geochemical constituents and also with petrographic elements has been discussed. This would help in planning the strategy for their utilization.

2 Geological setting

The sedimentary tract of Rajasthan is spread over a large area of 120000 km² and forms the eastern flank of Indus shelf. The entire sedimentary tract has been sub-divided into four basins which includes- (i) Palana-Nagaur basin, (ii) Jaisalmer basin, (iii) Barmer basin, and (iv) Sanchar basin (Jodha 2008). The present investigation has been carried out on Kasnau-Matasukh lignites. These lignites

occur in Palana-Nagaur basin which is an E-W trending elongated basin. It extends for 200 km in length and 50 km in width. Kasnau-Matasukh block is located in Jayal Tehsil of Nagaur district. Nagaur basin has several disconnected small basins of Palana Formation indicating undulating paleotopography. Nagaur basin is linked to a 5–6 m wide channel and hence known as link basin (Lal and Regar 1991). Though the structural features indicate a low tectonic disturbance in the area, gravity survey has indicated the presence of gravity low of 3–4 km width which occurs in NW–SE direction indicating the presence of link channel broadening towards SE. The magnetic survey of the area also substantiates the subsurface structures delineated through gravity survey and show NW–SE elongation of magnetic contour (Lal and Regar 1991). Geological work in and around Nagaur was initially taken up by Blanford (1876) who correlated the Jodhpur set with Vindhyan because of their closer resemblance. The interpretation of exploration data of Nagaur basin has been incorporated in the reports of Mukhopadhyay (1974–1975), Munshi (1975–1977), Faruqi (1978–1979, 1982–1983).

The lignite bearing Lower Tertiary sediments of Palana Formation are deposited unconformably over the Nagaur Formation. Presence of one lignite seam has been identified in the block which is intersected by a number of dirt bands. The Tertiary sequence of rocks comprises of three Formations which include Palana, Marh and Jogira in ascending order (Jodha 2009). The area has scanty outcrops. The lignite occurrences in the Nagaur basin are associated with Palana Formation of Paleocene age. The seams have been reported between 50 m and 150 m depths in the Palana Formation (Jodha 2009). Based on the recovered palynomorphs comprising pteridophytes, angiosperms, algae and fungi, Kulshrestha et al. (1989) and Shah and Kar (1971) have given Paleocene age to this Formation. The general stratigraphic succession of the rocks in the basin is given in Table 1 and the general geological map is shown in Fig. 1. The litholog (after Ghose 1983) and megascopic profile of the Kasnau-Matasukh lignite seam, prepared for this study, is shown in Fig. 2a while geological section is shown in Fig. 2b.

3 Method of study

Lignite samples have been collected from Kasnau-Matasukh mine of Nagaur basin of Rajasthan (Fig. 1) following pillar sampling method (Schopf 1960) so that full lignite seam thickness may be reconstructed in the laboratory. The samples have been crushed and reduced in quantity through quartering and coning to prepare eight composite samples which were subjected to various analyses. The samples were ground to pass 18 mesh size to prepare polished

Table 1 General stratigraphic succession in the Bikaner-Nagaur basin, Rajasthan (after Ghose, 1983)

Age	Formation	Lithounits	Thickness (m)
Pleistocene to recent	Kolayat formation	Sand and sandy alluvium	5–11
		Ironstone nodule, sandy calcareous grit kankar, gypsite, Ferruginous band, semi-consolidated conglomerate	1–2
		Erratic boulder of quartzite	?
Unconformity			
Early to middle eocene	Jogaria formation (Calcareous facies)	Shaly and marly limestone with foraminifers (<i>Alveolina</i> , <i>Discocyclina</i> <i>Nummulities</i>)	5–10
		Unfossiliferous, white clayey marl	1
		Dirty brown impure limestone with broken shells of <i>ostrea</i> and foraminifers (<i>Assilina</i>)	1.5
		Fuller's earth with shale partings having casts of lamellibranchs and gastropods	14
		Cream and yellowish white limestone full of smaller foraminifers (<i>Nummulites</i> and <i>Assilina</i>) with a thin band of fuller's earth (1–2 m) near base	75
		Yellow shales ochers, marl, etc. with smaller foraminifers (<i>Nummulites</i> , <i>Assilina</i>)	20
Angular unconformity			
Late paleocene (?)	Marh formation (Arenaceous facies)	Upper clay horizon with one clay bed	3–10
		Ferruginous sandstone, gritty sandstone and sugary sandstone with white glass sand (local)	60
		Middle clay horizon with five clay beds and sandstone partings	50
		Ferruginous sandstone, gritty sandstone, grit, siltstone	70
		Lower clay horizon with one clay bed	1–3
		Ferruginous sandstone, gritty sandstone, various siltstone with leaf impressions (base not exposed)	20
(?) Gradational contact			
Early paleocene (?)	Palana formation (Carbonaceous facies)	Fine grained sandstone Carbonaceous shale and lignite	?
Base not encountered			

mounts for petrography, while they were further ground to pass 70 mesh size for various chemical analyses like proximate, ultimate, rock–eval pyrolysis and major/minor/trace element analyses. Maceral analysis has been carried out to see the distribution of huminite, liptinite and inertinite group macerals. This is performed under reflected light using a Leitz Orthoplan-Pol Microscope equipped with Wild Photoautomat MPS 45 in the Coal and Organic Petrology Laboratory, Department of Geology, Banaras Hindu University. The line-to-line and point-to-point spacing was maintained at 0.4 mm and more than 600 counts have been taken on each sample following the methodology given by Taylor et al. (1998); huminite macerals have been termed and described as per ICCP-1994 (Sýkorová et al. 2005) while ICCP (2001) has been followed for inertinite macerals. The vitrinite/huminite reflectance (VRo) was measured at National Metallurgical Laboratory, Jamshedpur following ISO 7404-5:2009 (standard used: spinel, yag-yittrium, aluminium garnet, zirconia). On each sample, a minimum of 200 measurements

were taken. The proximate analysis has been carried out as per BIS (2003), while the elemental analysis (C, H, N, O, and S) has been performed at CMPDI, Ranchion Elementar Analysensysteme-Vario-III as per ASTM D5373-08.

The Pyrolysis has been carried out on high precision Rock-Eval-6 (make Vinci Technologies, France) at R & D department, Oil India Ltd, Duliajan (Assam) on fourteen lignite samples (represented as four composite samples). This is programmed pyrolysis system and is ultimate to know the source rock potential for hydrocarbon. The significance of this technique is that the coal samples, as such, are analyzed to know the various components. The analysis is performed under controlled temperature and coal samples are heated in absence of oxygen. The produced compounds are quantitatively assessed. During heating the oxygenated compounds released from mineral matter, present in coal, are excluded. The quantitative measurement of various fractions of volatile/non volatile organic compounds, source rock potential and the degree of maturation of lignites samples is obtained through this

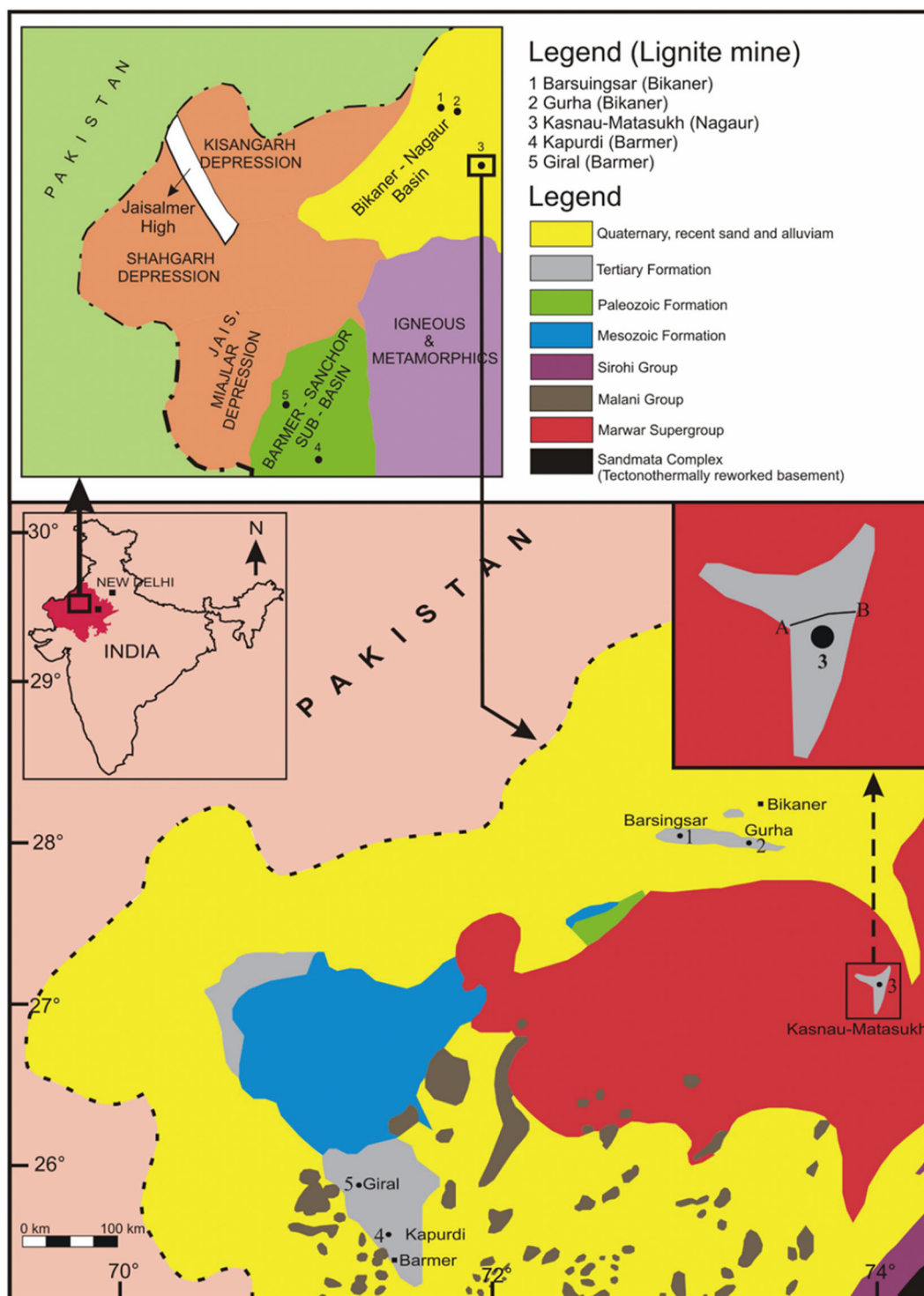


Fig. 1 Geological map of Nagaur basin (after Roy and Jakhar 2002) and location of Kasnau-Matasukh lignite

analysis. The pyrolysis of Kasnau-Matasukh lignite samples has been carried out following the procedures of Espitalié et al. (1977, 1984, 1986). The samples were heated in an open pyrolysis system under non-isothermal condition and the recorded FID signal is divided in two

surfaces, S_1 and S_2 , which are expressed in mg HC/g of coal. The method gets completed by combustion (oxidation) of the residual rock recovered after pyrolysis at 850 °C under nitrogen. This is required to avoid incomplete combustion. The released CO and CO₂ are monitored

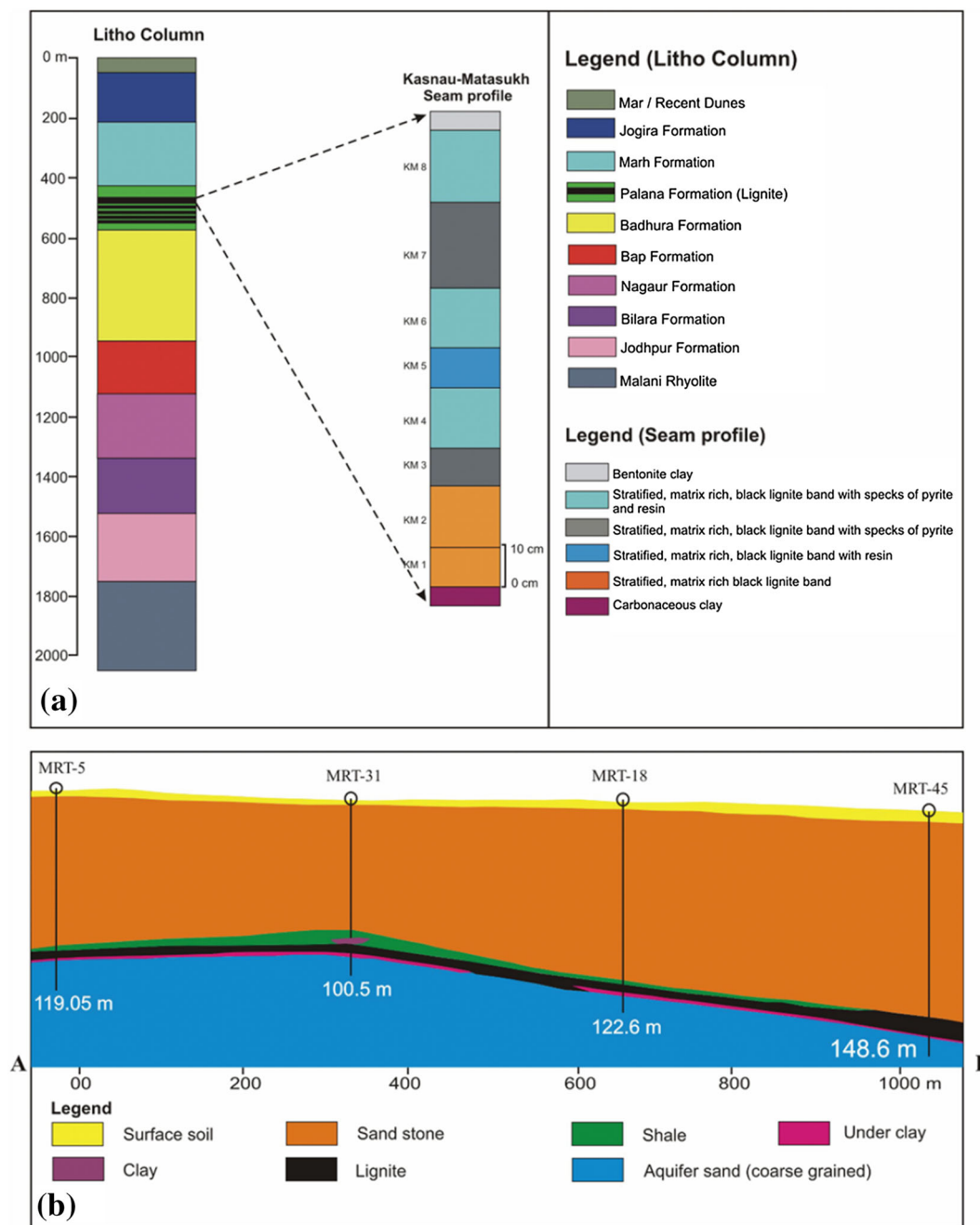


Fig. 2 Lithology (after Ghose 1983), megascopic profile of the Kasnau-Matasukh lignite seam showing various bands (a) and geological section (b) which is redrawn after Gowrisankaran et al. (1987)

online through an infra-red cell. This complementary data acquisition helps in the determination of total organic carbon (TOC) and total mineral or inorganic carbon (TMC or TIC).

The elements Fe, Ca, Mg, Mn, K, Na, Cu, Co, Ni, Cr, Zn, Pb and Ash have been determined on 'whole coal samples' in the department of Botany, BHU, Varanasi. For the determination of these elements the coal samples have been digested with 2.5 mL HNO_3 and HClO_4 in 10:1 ratio on hot

water plate following the method of Eaton et al. (1995). The mixture is then filtered using Whatman filter paper (No. 41) and the digested samples are rinsed with 1% Conc. HNO_3 . It is then transferred in a separate test tube and the volume is made up to 20 mL. The digested samples have been used for analyzing the concentrations of various elements under Atomic Absorption Spectrophotometer (AAS, Model Perkin Elmer Analyst 800) and the standard used in the analysis were Accu Standard solutions obtained from

Merck, KGaA, Darmstadt, Germany. Data of major, minor and trace elements are mean of three independent observations in the present paper. The measured values have shown relative standard deviations less than 5 % for all the elements in the analyzed samples.

Fourier Transform Infrared spectra have been recorded by FTIR spectrophotometer (PerkinElmer Spectrum version 10.03.05) using KBr pellets (transmission mode) in the Department of Chemistry, Banaras Hindu University. Coal: KBr mixture at 1:100 ratio has been used and 20 number of scans have been taken with a spectral resolution of 4 cm^{-1} at a range of $400\text{--}4000\text{ cm}^{-1}$. X-ray diffraction data have been obtained with the help of computer controlled X-ray Diffractometer Panalytical X'Pert High Score (Plus) v3 database in the Department of Geology, Banaras Hindu University. The operating parameters, in the present study are: start angle- 2° ; target- Cu $K\alpha$ radiation; stop angle- 60° ; step size- 0.0250; and 2 theta configuration.

4 Result and discussion

4.1 Petrographic characteristics

These lignites are stratified in nature and are of black color. Huminite is the main component in these lignites (Singh et al. 2015a, b) which is formed due to anaerobic preservation of lignocellulose material in the mire (Sýkorová et al. 2005). Liptinite and inertinite macerals occur in low concentrations. Huminite (83.9 %–92.5 %; av. 87.3 % mineral matter free basis) is largely contributed by detrohuminite and telohuminite. Detrohuminite is represented by densinite (19.2 %–42.5 %; av. 31.7 % mineral matter free basis) and attrinite (0 %–13.3 %; av. 5.9 % mineral matter free basis) while telohuminite is represented by ulminite-A (24.9 %–38.6 %; av. 30.5 % mineral matter free basis), ulminite-B (13.4 %–29.1 %; av. 18.2 % mineral matter free basis) and textinite which occurs in very low amount (<1 %). Liptinite group (5.7 %–13.2 %; av. 10.9 %) and inertinite group (0.2 %–4.0 %; av. 1.9 %) are low in concentrations (Table 2). Mineral matter ranges between 3.5 and 12.0 (av. 7.7 %). The vertical variation of group macerals and mineral matter from base of the seam is shown in Fig. 3. Though, there is no specific trend of variation, yet huminite shows a high concentration at the upper part while liptinite shows a reverse trend. Inertinite is less at the bottom. Mineral matter is more towards the upper part of the seam. The variation has environmental implications. The clastic mineral matter relates directly to water cover in the basin and, therefore, it increases with increase in the water cover during the formation of upper part of the lignite seam. This is also supported by the occurrence of high concentration of huminite group macerals during this period.

4.2 Chemical attributes

These lignites have high volatile matter content (52.6 %–67.0 % daf basis; av. 58.3 %) with moderate ash yield (3.0 %–18.2 %; av. 8.4 %). The ultimate analysis (av. values on daf basis) shows that these lignites contain 54.0 % carbon, 5.4 % hydrogen, 0.8 % nitrogen, 35.9 % oxygen and 3.8 % sulfur (Table 2). The vertical variation of the chemical components along the seam profile is shown in Fig. 4. Volatile matter shows an increasing trend towards upper part of seam while fixed carbon shows a reverse trend. Carbon and sulfur show an increasing trend towards upper part while other ultimate components like hydrogen, nitrogen and oxygen do not show any definite trend. Variable concentrations and dimensions of undecomposed, partly decomposed and completely decomposed wood have been noticed in the Kasnau-Matasukh lignites. This appears to have affected the proximate and ultimate composition of this lignite from bottom to top because these three components have variation in the organic geochemical constitution.

4.3 Hydrocarbon potential

These lignites of Nagaur basin have attained a thermal maturity indicated by vitrinite reflectance (VRo) between 0.23 % and 0.30 % (Table 3) which put them as 'low rank C' coals as per ISO-11760 (2005). The analytical results of Rock-Eval pyrolysis of Kasnau-Matasukh lignites show that S_1 values (free hydrocarbon distilled out of samples at initial heating of 300°C) vary from 1.16 to 2.83 mg HC/g. Considering 1 mg HC/g as its cut-off value, this lignite may be considered as a good source rock. Similarly, S_2 values (hydrocarbons generated through thermal cracking which actually indicate the quantity of hydrocarbons that the lignite may potentially produce) are many-fold higher than the free hydrocarbons (already generated oil in the lignite and occur as free hydrocarbons in lignite samples) and it varies from 48.65 to 87.84 mg HC/g (av. 68.99 mg HC/g). Taking 5 as its cut-off value, it also indicates a good source rock for hydrocarbon generation. The S_3 values vary from 24.34 to 29.89 mg CO_2 /g and represent the trapped carbon-dioxide which is released during the pyrolysis up to a temperature of 390°C . This is also proportional with the oxygen present in the Kasnau-Matasukh lignites of Nagaur basin. Total organic carbon (TOC) content of these lignite samples exhibits a wide range from 3.27 % to 43.92 % with an average of 31.08 % while the total inorganic carbon (TIC) values also have the similar trend and the value ranges from 1.85 % to 2.73 %.

The vertical variation of the Rock-Eval data from the base of the lignite seam is shown in Fig. 5. It is evident from this figure and the data (Table 4), that S_1 value is low

Table 2 Petrographic and chemical constituents in the Kasnau-Matasukh lignites

Sample number	Megascopic characteristics	Petrographic components (vol%)				Proximate components (wt%)			Ultimate components (wt%)				H/C	O/C	
		Huminite	Liptinite	Inertinite	Mineral matter	Air dried	Ash	Dried ash free	Dried ash free						
									Volatile matter	Fixed carbon	C	H			N
(Top) KM 8	Stratified, matrix rich, black inhomogeneous lignite with presence of pyrite and resin	82.7 (92.5)	5.1 (5.7)	1.6 (1.8)	10.6	11.5	67.0	33.0	52.6	5.6	0.8	36.5	4.4	1.27	0.52
KM 7	Stratified, matrix rich, black inhomogeneous lignite with presence of pyrite	78.2 (84.8)	12.1 (13.1)	2.0 (2.1)	7.8	7.5	54.6	45.4							
KM 6	Stratified, matrix rich, black inhomogeneous lignite with presence of pyrite and resin	76.2 (86.6)	10.8 (12.3)	1.0 (1.1)	12.0	18.2	58.4	41.6							
KM 5	Stratified, matrix rich, black inhomogeneous lignite with presence of resin	82.5 (87.0)	10.8 (11.4)	1.5 (1.6)	5.2	4.9	63.3	36.7	53.7	5.1	0.8	36.5	3.8	1.14	0.51
KM 4	Stratified, matrix rich, black inhomogeneous lignite with presence of pyrite and resin	80.9 (89.3)	8.5 (9.4)	1.2 (1.3)	9.5	9.6	57.5	42.5							
KM 3	Stratified, matrix rich, black inhomogeneous lignite with presence of pyrite	80.6 (86.9)	8.4 (9.0)	3.7 (4.0)	7.2	7.1	58.2	41.8							
KM 2	Stratified, matrix rich, black inhomogeneous lignite	80.9 (83.9)	12.8 (13.2)	2.8 (2.8)	3.5	3.0	52.6	47.4							
(Bottom) KM 1	Stratified, matrix rich, black inhomogeneous lignite	82.5 (87.2)	11.9 (12.6)	0.2 (0.2)	5.4	5.1	54.3	45.7	55.7	5.6	0.9	34.6	3.2	1.19	0.47
Mean		80.6 (87.3)	10.1 (10.8)	1.7 (1.9)	7.7	8.4	58.3	41.8	54.0	5.4	0.8	35.9	3.8	1.20	0.50

Note Values in parenthesis are recalculated on m.m.f. basis

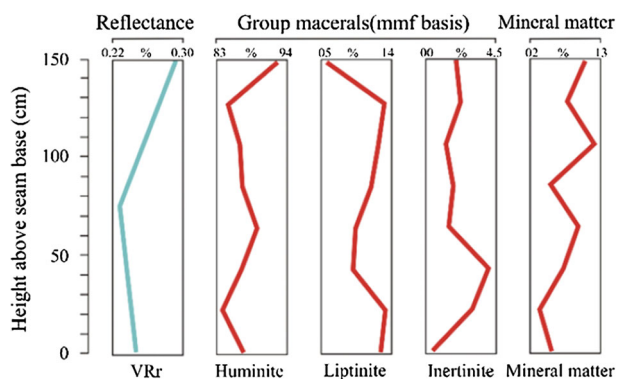


Fig. 3 Vertical variation of group macerals and mineral matter along lignite seam profile

at the bottom while S_2 value is more at the middle part of the seam which decreases towards the top as well as towards the bottom part (Table 4; Fig. 5). Total organic carbon content has an increasing trend towards the mid of the seam while decreases at the upper part. The total inorganic carbon (TIC) values also have the similar trend and are derived from the carbonates. In this seam the sulfur content (varies from 3.2 % to 4.4 %) maintains a strong negative correlation ($r = -0.81$; P value = 0.399) with the TOC content and also with TIC ($r = -0.78$; P

value = 0.433). The organic matter (OM, obtained by deducting ash content from hundred) shows a variation from 81.8 % to 97 %.

Coal acts as good source rock for hydrocarbon generation. The H/C ratio, in 0.8–0.9 range, is a good indicator of a source rock having hydrocarbon potential (Powell and Boreham 1994). Certain coals with low liptinite content have hydrogen-rich vitrinite which generates oil (Bertrand 1989; Newman et al. 1997; Petersen et al. 2000; Singh 2012; Singh et al. 2013). The generated hydrocarbon products have a finite storage capacity and until this capacity is exceeded, no oil expulsion takes place (Powell 1978; Mc Auliffe 1979; Durand 1983; Tissot and Welte 1984; Inan et al. 1998). Singh (2012) and Singh et al. (2016) have studied the hydrocarbon potential of lignites of Cambay basin and Bikaner basin (India) respectively while Singh et al. (2013) investigated the sub-bituminous coals of east Kalimantan (Indonesia) for its liquid hydrocarbon potential. The cross plot of hydrogen index (HI) with oxygen index (OI) and T_{max} of Kasnau-Matasukh lignite (Fig. 6) indicates its immaturity. This lignite falls closer to the zone of organic rich type-III kerogen which is formed under topogenous condition as also revealed by a cross plot between total organic carbon (TOC) and sulfur content (Fig. 7). This plot is proposed by Jasper et al. (2010) who

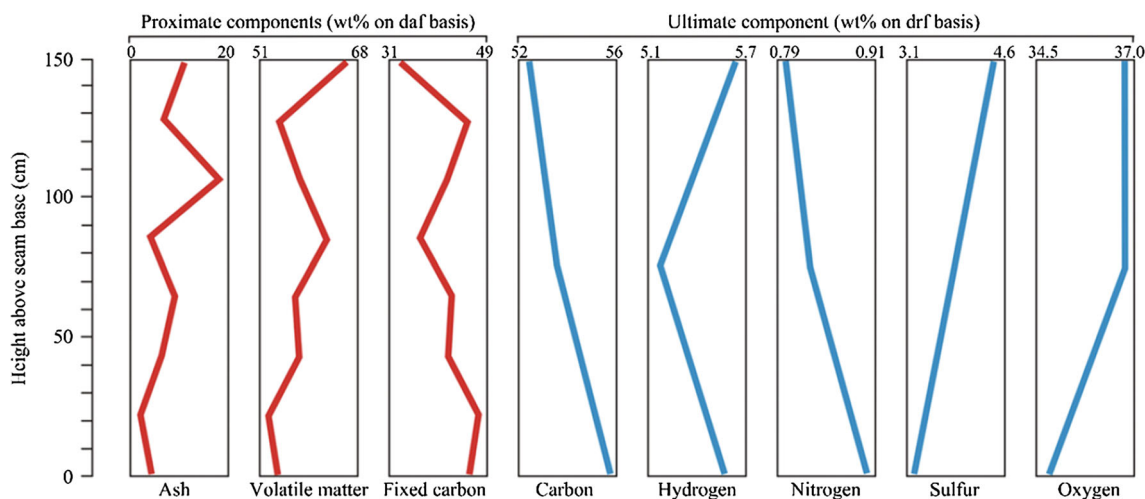


Fig. 4 Vertical variation of geochemical components from base of the lignite seam

Table 3 Vitrinite/huminite reflectance data in the Kasnau-Matasukh lignites

Sample number	Mean value	SD	Variance	Maximum	Minimum
KM-1	0.25	0.06	0.00	0.41	0.09
KM-5	0.23	0.06	0.00	0.40	0.09
KM-8	0.30	0.07	0.00	0.46	0.15
Mean (Mine)	0.26	0.06	0.00	0.43	0.11

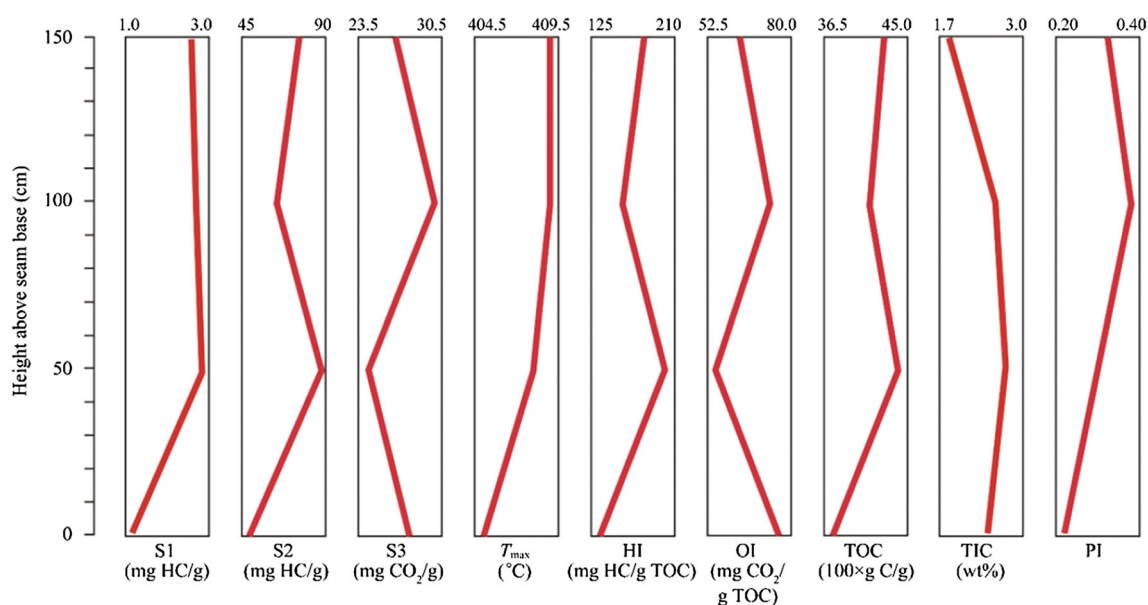


Fig. 5 Vertical variation of Rock–Eval components from base of the lignite seam

Table 4 Rock–Eval data in the Kasnau-Matasukh lignites

Sample number	S ₁ (mg HC/g)	S ₂ (mg HC/g)	S ₃ (mg CO ₂ /g)	T _{max} (°C)	HI	OI	TOC(%)	TIC	PI
KM-8	2.58	75.76	26.59	409	179	63	42.3	1.85	0.03
KM-5	2.69	63.71	29.89	409	157	73	40.67	2.57	0.04
KM-3	2.83	87.84	24.34	408	200	55	43.92	2.73	0.03
KM-1	1.16	48.65	27.81	405	133	76	36.46	2.45	0.02
Mean	2.32	68.99	27.16	407.75	167.25	66.75	40.84	2.40	0.03

Note HI hydrogen index, OI oxygen index, TOC total organic carbon, TIC total inorganic carbon

have grouped the coals in three domains. The coal, in Group-A, is for high TOC and low sulphur content (<2 %) which evolves under ombrogenous mires having raised bogs. Such mires are fed by rainwater. Group-C is characterized by coals with high sulphur content and is formed under topogenous mires. Group-B, however, shows an intermediate condition. On this plot the Kasnau-Matasukh lignite falls under Group-C which is characterized by coals having high sulphur content and is formed under topogenous mires as per Jasper et al. (2010). The H/C atomic ratio of 1.2 (Table 2), and high concentration (>80 %) of reactive macerals (huminite + liptinite), in Kasnau-Matasukh lignites (Table 2), are indication of their good hydrocarbon potential as per Cudmore (1977) and Davis et al. (1976). These lignites have potential of generating mainly gaseous hydrocarbons. The cross plot between vitrinite reflectance and HI (Fig. 8) also indicates that these lignites are mainly gas prone. The details of the maturity and oil generating potential of the lignites of entire Bikaner-Nagaur basin have been discussed in detail by Singh et al. (2016).

4.4 X-ray diffraction (XRD) and Fourier transform infrared (FTIR) studies

XRD spectra of whole coal and low temperature ash samples of the Kasnau-Matasukh lignites are shown in Fig. 9a, b. The minerals in these coals were identified by comparing 'd' values as per Lindholm (1987). The common minerals identified from XRD spectrum of whole coal sample are biotite, gypsum, chlorite, goethite/laumontite, quartz, barite, dolomite, haematite and marcasite. The minerals identified in the low temperature ash include goethite/laumontite, anorthite, quartz, haematite and mixed clay. Kaolinite, illite and chlorite are the major mixed clay minerals. Haematite, goethite and marcasite are major iron containing minerals while gypsum, dolomite and laumontite are calcium rich. FTIR spectra are useful for the identification of minerals associated with the coal structures (Karr 1978). The peaks in FTIR spectra of coal between 1100 and 400 cm⁻¹ are of clay minerals such as quartz, kaolinite, illite and montmorillonite groups. The

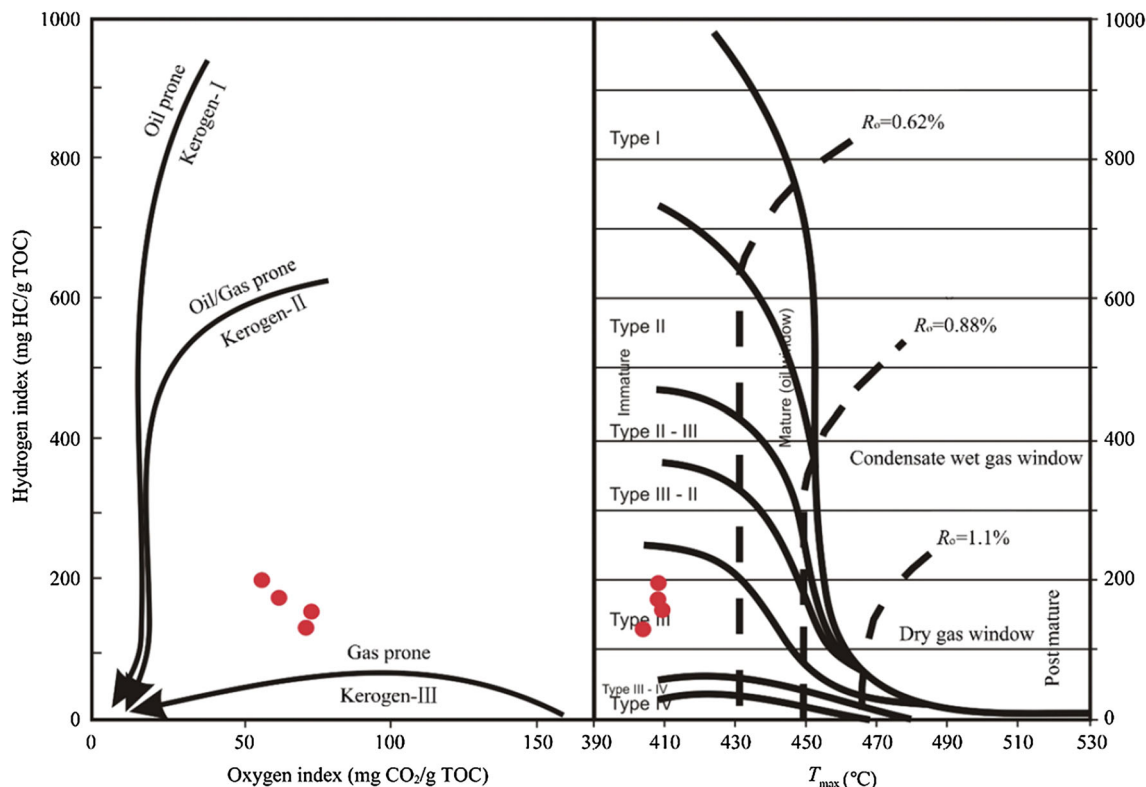


Fig. 6 Cross plot of hydrogen index (HI) with oxygen index (OI) (Krevelen 1961) and T_{max} (°C) (after Koeverdon et al. 2011)

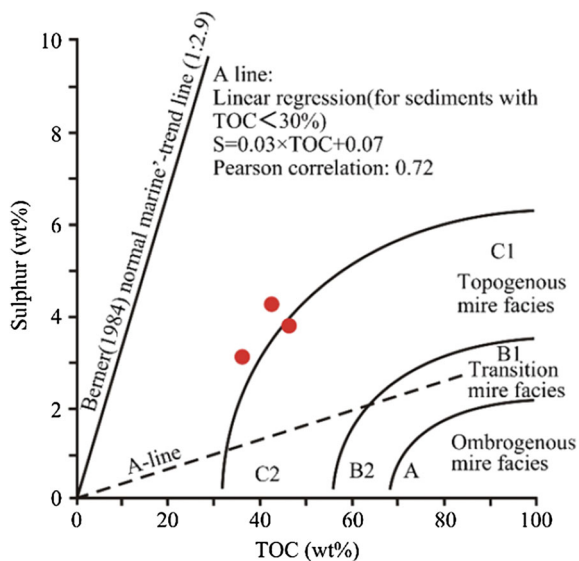


Fig. 7 Cross plot between Sulfur and total organic carbon (TOC) (after Jasper et al. 2010)

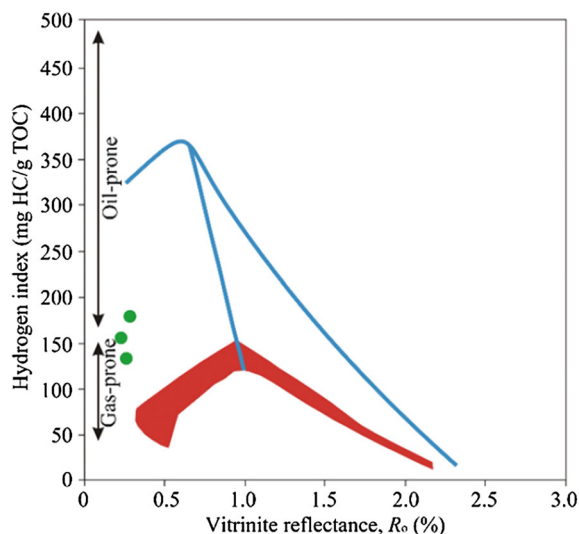


Fig. 8 Cross plot between Hydrogen Index (HI) and Vitrinite Reflectance (VRo) (after Petersen 2005)

different absorption peaks with their bonds and functional groups are furnished in Table 5 and shown in Fig. 9c. The broad absorption bands in coal ranging from 3618 to 3628 and 3696 to 3699 cm^{-1} belong to clay minerals (kaolinite and illite). The absorption bands at 3694.90 and

2920.08 cm^{-1} in the coals are due to O–H groups while the absorption bands at 3397.37 cm^{-1} are due to O–H and N–H groups. Strong aliphatic absorptions are observed at 2920–2850 cm^{-1} . The intensity of peaks at 2920 cm^{-1} indicates the presence of long aliphatic chains in the Kasnau-Matasukh lignites. Low intensity aromatic bands were

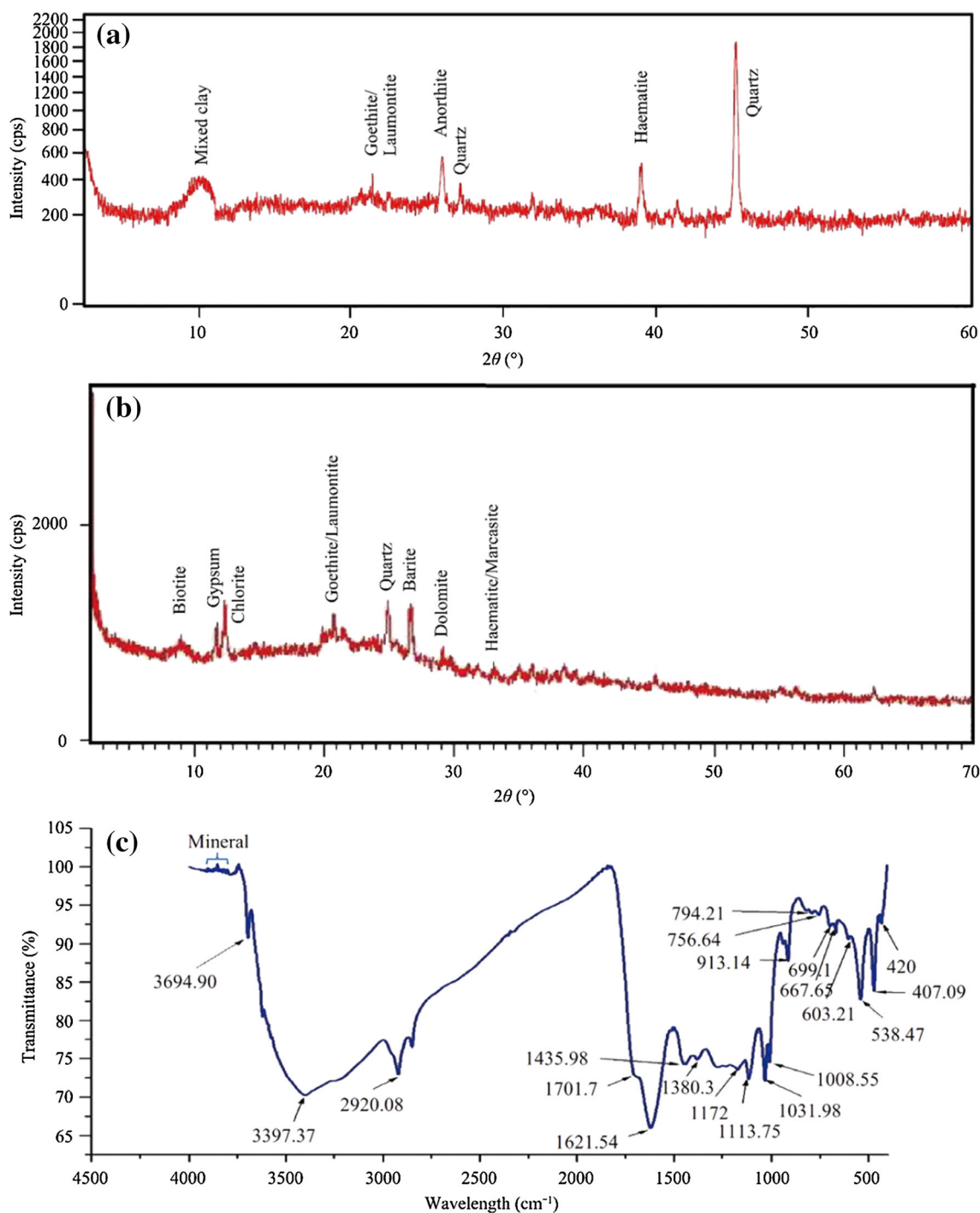


Fig. 9 X-ray diffraction pattern and FTIR spectrum of the lignite samples of Kasnau-Matasukh

observed in $900\text{--}700\text{ cm}^{-1}$ regions in these lignites. The peak 1701.7 cm^{-1} appears due to the presence of carbonyl ($\text{C}=\text{O}$) group while the peak 1621.54 cm^{-1} appears due to the presence of 1° amines (N-H). The peak 1435.98 cm^{-1} indicates the presence of aromatics (C-O stretch in ring). The peaks near 1172 and 1113.75 cm^{-1} indicate the presence of aliphatic amines (C-N). The oxygen containing

functional groups are phenols, alcohols, ethers, carboxylic acid and carbonyls. The region of $1000\text{--}1300\text{ cm}^{-1}$ in the spectra is of C-O bonds. The weak band at 756.64 cm^{-1} could be due to C-Cl bond while the weak band at 699.1 cm^{-1} may be due to $\text{C}\equiv\text{C-H}$; C-H bonds. The peak at 667.65 cm^{-1} may be due to $\text{C}\equiv\text{C-H}$; C-H bonds. The weak bands at 603.21 and 538.47 cm^{-1} is due to C-Br

Table 5 Band assignment of the most prominent peaks in the FTIR spectra of Kasnau-Matasukh lignite

Bands (cm ⁻¹)	Assignments
466–470 and 528–535	Clay and silicate minerals
669	Aromatic out of plane bebding (?)
690–515	Alkyl halides (C–Br stretch)
700–610	Alkynes (–C≡C–H: C–H bend)
725–720	Alkanes (C–H rock)
850–550	Alkyl halides (C–Cl stretch)
900–675	Aromatics (C–H “oop”)
910–665	1°, 2° amines (N–H wag)
950–910	Carboxylic acids (O–H bend)
1032–1047 and 1115–1118	Silicate (Si–O), C–O and C–O–R structures
1250–1020	Aliphatic amines (C–N stretch)
1300–1150	Alkyl halides C–H wag (–CH2X)
1370–1350	C–H rock (alkanes)
1500–1400	Aromatics C–C stretch (in–ring)
1612–1622	C=C aromatic stretching and C=O conjugated
1650–1580	1° amines (N–H bend)
1710–1665	α,β-unsaturated aldehydes, ketones (C=O stretch)
2851	Aliphatic symmetric –CH2 stretching
2918–2926	Aliphatic asymmetric –CH2 stretching
3000–2850	Alkanes (C–H stretch)
3402–3416	–OH stretching
3400–3250	1°, 2° amines, amides (N–H stretch)
3640–3610	Alcohols and phenols (O–H stretch, free hydroxyl)
3618–3628 and 3696–3699	Clay minerals (kaolinite and illite)

bond. The present study is in agreement with the studies of Georgakopoulos et al. (2003), Saikia et al. (2007) and Zodrow et al. (2010).

4.5 Geochemistry of major/minor and trace elements

The study of trace elements in coal is being given more impetus during last few decades owing to their environmental implications. Mode of occurrence of major, minor and trace elements in coal may be known through direct and indirect methods (Eskenyzy and Stefanova 2007). In the lignite samples of Kasnau-Matasukh, the mode of occurrence of elements has been studied through indirect method. Here, correlation coefficients of the elements with ash yield, petrographic content and also among themselves have been calculated.

The concentration of elements in the analysed samples has been compared with the world average in lignite. As

Table 6 Major, minor and trace element contents (in ppm) in the Kasnau-Matasukh lignites

Metal	WCBC*	Sample number							
		KM-1	KM-2	KM-3	KM-4	KM-5	KM-6	KM-7	KM-8
Fe	10000	388.00	414.00	392.00	61.20	432.00	418.00	472.00	506.00
Cu	15	342.00	344.00	374.00	232.00	280.00	432.00	1078.00	145.80
Co	4.2	2.00	2.40	1.70	1.30	2.62	4.80	1.44	2.28
Ni	9	5.86	5.52	6.42	2.90	3.58	7.30	4.88	4.38
Cr	15	10.24	6.16	5.72	6.54	5.52	7.60	12.60	6.58
Zn	18	15.56	9.52	60.00	10.28	10.68	12.30	13.78	9.58
Pb	6.6	3.06	4.00	2.42	2.76	3.46	7.62	3.90	4.72
Mg	200	46.40	47.00	47.20	47.20	46.80	46.60	47.20	46.80
Cd	0.24	0.52	0.64	0.64	0.74	0.62	0.50	0.92	0.74
Na	200	40.00	42.80	49.20	34.40	87.60	187.60	41.60	51.60
K	100	7002.00	6398.00	7812.00	6332.00	3768.00	7172.00	6742.00	5064.00
Ca	10000	1558.00	1264.00	492.00	364.00	2020.00	578.00	2220.00	2300.00
Mn	100 ± 6	8.16	13.18	12.88	12.38	14.20	37.20	17.60	49.40

Note * World average values (in brown coal) of Ca, Fe, Mn, Na, K and Mg are after Valkovic (1983) and Clarke values (in brown coal) of rest of the elements is after Ketris and Yudovich (2009)

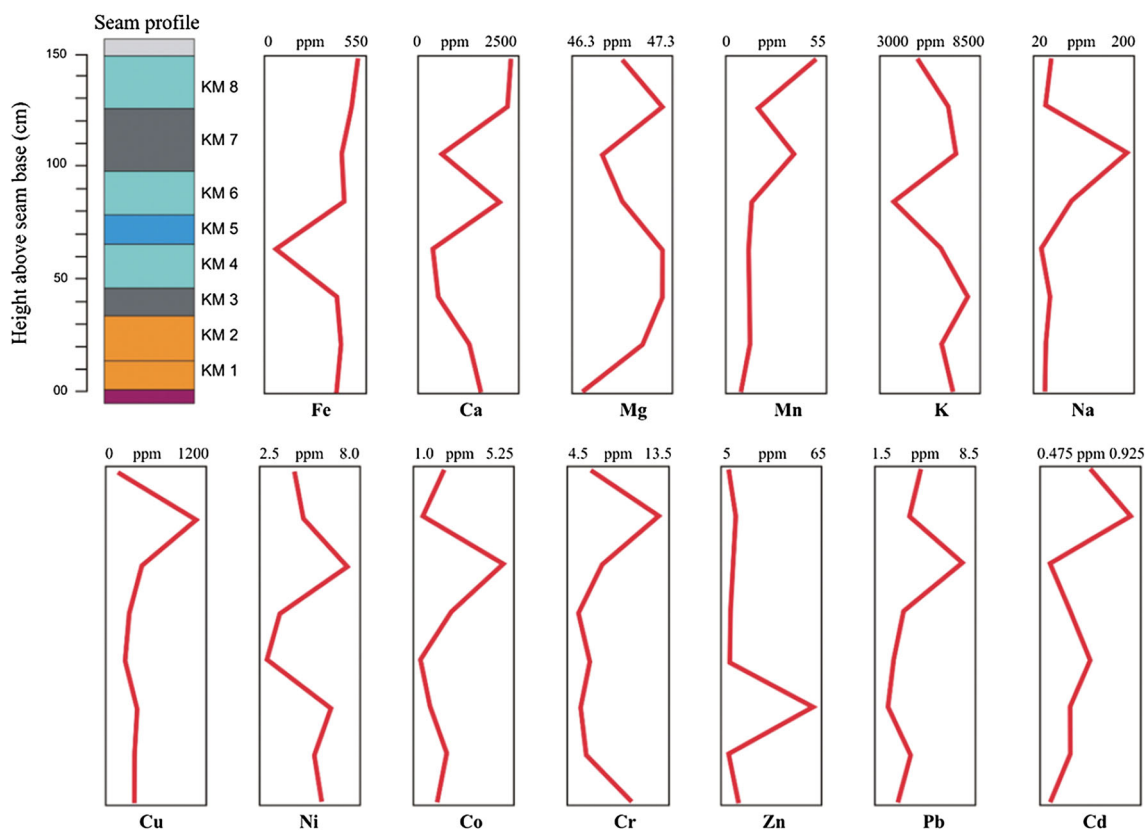


Fig. 10 Vertical variation of the concentration of major/minor and trace elements along the lignite seam profile

Table 7 Concentration of various pyrite forms identified under microscopy in the Kasnau-Matasukh lignites

No.	Sample number	Disseminated	Discrete	Framboidal	Massive	Fissure
1	KM-8	25.0	37.5	6.3	18.8	12.5
2	KM-7	11.1	22.2	44.4	11.1	11.1
3	KM-6	46.2	30.8	23.1	0.0	0.0
4	KM-5	35.7	21.4	21.4	14.3	7.1
5	KM-4	28.6	28.6	14.3	0.0	28.6
6	KM-3	50.0	16.7	16.7	16.7	0.0
7	KM-2	33.3	33.3	0.0	33.3	0.0
8	KM-1	100.0	0.0	0.0	0.0	0.0

we can see from the Table 6 the concentration of Cu is very high in all the bands and over 70 times, in KM-7 band, as compared to world average in brown coals. Similarly, Cd is 2–3 times high in almost all the bands while Zn is high in KM-3 band. Rests of the elements have a normal concentration in Kasnau-Matasukh lignites. The vertical variation of various major/minor and trace elements is shown in Fig. 10 along the lignite seam profile. Though, there is not a prominent trend of distribution of these elements yet the concentration of elements like Mn, Na, Cu, Ni, Co, Cr, Pb

and Cd is higher towards the upper part of the seam as revealed in Fig. 10. Sulfur concentration is high in Kasnau-Matasukh lignites. Pyrite is formed, in coal, from H_2S and Fe in solution which involves bacterial reduction of SO_4 to H_2S at pH 7–4.5 (Ryan and Ledda 1997). It occurs in various forms in Kasnau-Matasukh lignites. As analyzed under microscope, disseminated pyrite in these lignites, dominates (av. 41 %) over the other pyrite forms and is followed by discrete pyrite grains (av. 23.8 %) and massive pyrite (av. 11.7 %) (Table 7). Some photomicrographs of

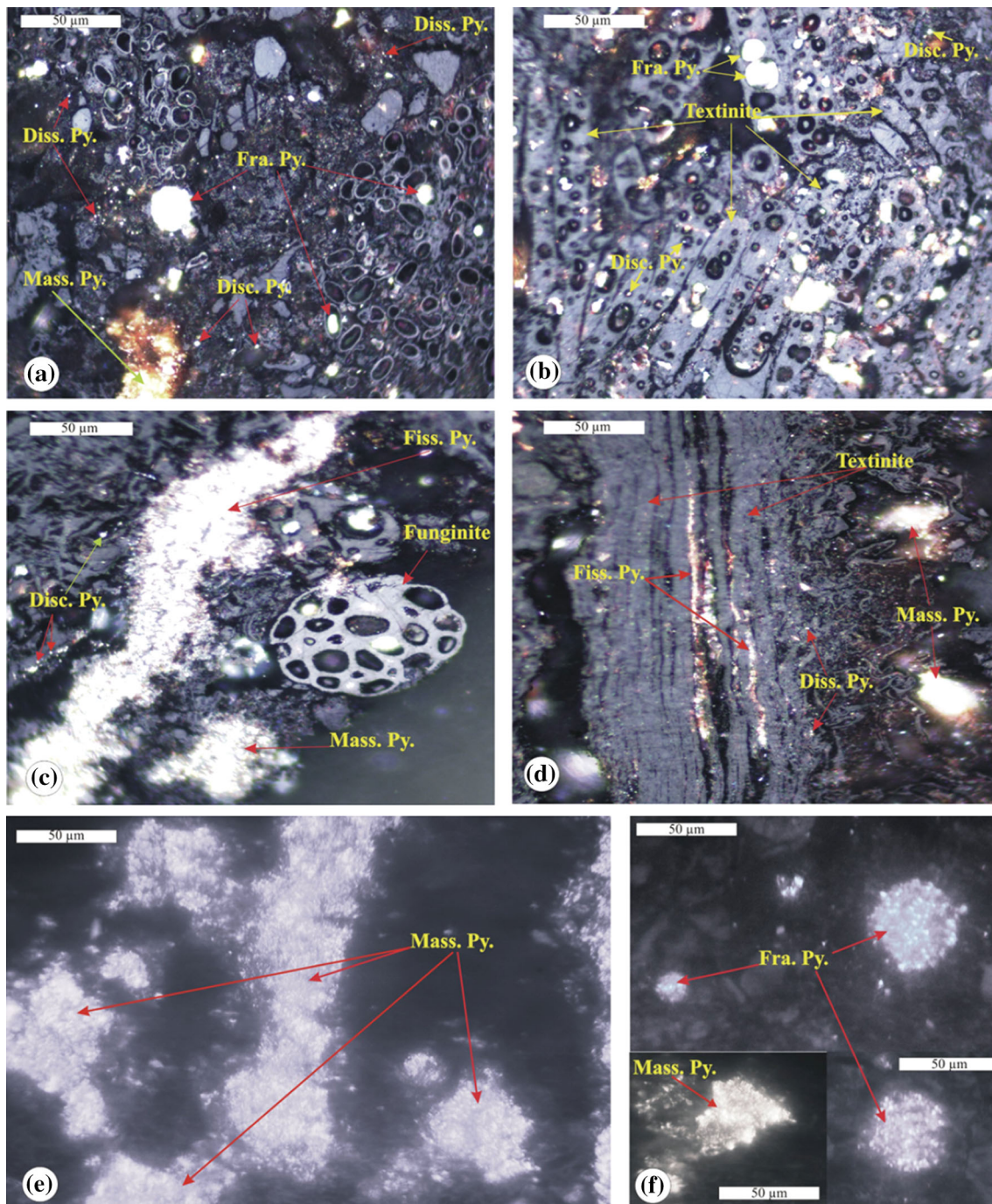


Fig. 11 Photomicrographs of minerals in the lignite samples of Kasnau-Matasukh; **a** framboidal pyrite, disseminated pyrite, discrete pyrite and massive pyrite; **b** framboidal pyrite and discrete pyrite occurring in textinite maceral; **c** massive pyrite, discrete pyrite, pyrite occurring as fissure and also in funginite maceral; **d** massive pyrite, disseminated pyrite, pyrite occupying the fissures in the lamellar textinite; **e** massive pyrite; **f** massive and framboidal pyrite; abbreviations: *Fra. Py.* framboidal pyrite, *Diss. Py.* disseminated pyrite, *Disc. Py.* discrete pyrite, *Mass. Py.* massive pyrite, *Fiss. Py.* fissure pyrite

pyrite of Kasnau-Matasukh lignites are shown in Fig. 11. The clustered framboidal pyrites are more common in the middle part of the seam while single framboids are more towards the upper part. Based on the values of correlation coefficient, preferential enrichment of Ni, Pb, and Co is

seen in pyrite. Finkelman (1994) also has reported the association of Co with sulfides, though, it is also found associated with clays and organic matter. Co may also occur as siegenite and cattierite (Dai et al. 2015a, b). Dale et al. (1999) have reported the occurrence of Co associated

Table 8 Correlation matrix between minor/trace, group maceral and type of pyrite in Kasnau-Matasukh lignite

Element	Fe	Ca	Mg	Mn	K	Na	Cu	Co	Ni	Cr	Zn	Pb	Cd	Ash	OM	Humi	Lipti	Inerti
Fe	1																	
Ca	0.7	1																
Mg	-0.3	-0.2	1															
Mn	0.4	0.3	-0.2	1														
K	-0.2	-0.6	0.14	-0.2	1													
Na	0.2	-0.3	-0.4	0.5	0.03	1												
Cu	0.3	0.3	0.3	-0.2	0.3	-0.03	1											
Co	0.3	-0.2	-0.6	0.5	-0.01	.952**	-0.2	1										
Ni	0.4	-0.3	-0.4	0.14	0.7	0.5	0.2	0.6	1									
Cr	0.2	0.4	-0.10	-0.12	0.3	-0.14	.805*	-0.2	0.2	1								
Zn	0.04	-0.4	0.3	-0.3	0.5	-0.13	0.02	-0.2	0.4	-0.2	1							
Pb	0.4	0.0	-0.4	.708*	0.03	.864**	0.07	.900**	0.5	0.06	-0.4	1						
Cd	-0.02	0.4	.727*	0.0	-0.12	-0.5	0.6	-0.6	-0.5	0.4	-0.09	-0.3	1					
Ash	-0.02	-0.3	-0.2	.728*	0.2	.744*	-0.03	0.6	0.3	0.001	-0.11	.786*	-0.2	1				
OM	0.02	0.3	0.2	-.728*	-0.2	-.744*	0.03	-0.6	-0.3	0.001	0.11	-.786*	0.2	-1.000**	1			
Humi	-0.15	0.12	-0.15	0.6	-0.3	-0.09	-0.6	-0.09	-0.4	-0.3	-0.09	-0.01	0.06	0.4	-0.4	1		
Lipti	0.07	-0.04	-0.2	-0.6	0.3	0.2	0.5	0.2	0.3	0.5	-0.2	0.14	-0.2	-0.3	0.3	-0.904**	1	
Inerti	0.2	-0.2	.707*	-0.11	0.2	-0.2	0.12	-0.3	0.2	-0.4	0.7	-0.3	0.3	-0.3	0.3	-0.3	-0.2	1

Humi huminite, *Lipti* liptinite, *Inerti* inertinite, *Py* pyrite, *Diss* disseminated, *Disc* discrete, *Fram* frambooidal, *Mass* massive, *Fiss* fissure

* Correlation is significant at the 0.05 level (2-tailed)

** Correlation is significant at the 0.01 level (2-tailed)

with silicates in Australian coals. Framboidal pyrite has shown preferential enrichment of Cu, Pb, Co, Cr, and Ni; disseminated pyrite shows an affinity with Ni and Co while discrete pyrite grains with Pb and Co. Similarly massive pyrite has a close affinity with Fe and Zn while pyrite occurring as fissure and crack fillings has affinity with Cd and Mg. Cadmium is normally associated with sphalerite (ZnS) though it is also found in other sulphides (Finkelman 1994). This has also been documented by Swaine (1990), Goodarzi (2002), and Dale et al. (1999). Ash yield shows a strong affinity with Mn ($r = 0.728$) and Na ($r = 0.744$) among the major elements and with Pb ($r = 0.786$) and Co ($r = 0.65$) among the trace elements. Eskenzy (2009) also reported a positive correlation of ash content with Mn and Co and observed the association of Pb with organic as well as inorganic fractions in Bulgarian coal. On the other hand inertinite maceral group has shown a strong affinity with Mg and Zn while huminite has a strong affinity with Mn, and liptinite relates well with Cu and Cr. These elements could either be associated with the organic molecules or with the minerals occurring as intergrown with the macerals. On the other hand some elements could be related to those minerals which occur as surface blanketing or as superficial mounting over the surface of the macerals (Singh et al. 2010). While working on Shenbei Tertiary lignites of China, Ren et al. (2004) reported Cr, Co, Ni, Cu, V and Zn to be associated with organic macromolecules and they have suggested their enrichment during coal-forming or early diagenesis process. Eskenzy and Stefanova (2007) believe that organically bound parts of the elements are generally higher in low-rank coals. Due to lack of evidence regarding mode of occurrence of Ni in coal, its relation is yet to be precisely established (Finkelman 1994; Riley et al. 2012). It may be organically bound or it could also be associated with sulfides. Dale et al. (1999) reported Ni from both monosulphides and organic matter. Ni relates with Na, K and Co in Kasnau-Matasukh lignites which is in agreement with the work of Singh et al. (2015a, b) on the nearby located Barsingsar and Gurhal lignites of Bikaner-Nagaur basin. KM-3, which is matrix rich stratified band, contains high concentration of Zn, Cu and Cd. Zinc is considered as a notorious contaminant and occurs in all coals in HCl soluble phase (Riley et al. 2012).

As revealed in the correlation matrix among the elements in the Kasnau-Matasukh lignites, Cd shows a strong affinity with Cu while Pb has a strong affinity with Co and Ni. Pb also has an affinity with sulfides especially pyrite. Cr relates strongly with Cu and occurs in sulphides while Co maintains a positive affinity with Na and Mn (Table 8).

5 Conclusion

1. These lignites are predominantly composed of huminite group of macerals while liptinite and inertinite macerals occur in less concentration. Huminite shows a high concentration at the upper indicating anaerobic degradation during that period. Mineral matter is more towards the upper part of the seam indicating a wet environment.
2. Volatile matter content is high while ash yield is moderate. Sulfur content of these lignites is moderately high. There is increase in volatile matter, carbon and sulphur contents towards the upper part of seam.
3. S_1 values are low at the bottom while S_2 values are more at the middle part of the seam and decreases towards the top as well as bottom. Total organic carbon content is more in the middle part of the seam and decreases towards the top. Study reveals that these lignites are type-III kerogen and are mainly gas prone.
4. XRD study reveals the presence of mixed clay minerals including kaolinite, illite and chlorite. The peaks in FTIR spectra between 1100 and 400 cm^{-1} further support the presence of these clay minerals.
5. The concentration of Cu is very high in all the samples and over 70 times in KM-7 band. Similarly, Cd is 2–3 times high in almost all the samples while Zn is high in KM-3 band. The concentration of elements like Mn, Na, Cu, Ni, Co, Cr, Pb and Cd is higher towards the upper part of the seam. Preferential enrichment of Ni, Pb, and Co is seen in pyrite.
6. Ash content shows a strong affinity with Mn, among the major elements, and with Co among the trace elements. On the other hand, inertinite maceral has an affinity with Mg and Zn while huminite with Mn, and liptinite with Cu and Cr. Cadmium shows a strong affinity with Mg and Cu while Pb has a strong affinity with Mn, Na, Co and Ni. Chromium relates strongly with Cu, Pb with pyrite, Co with Na and Mn; and Ni with Na, K and Co.

Nevertheless, the results warrant further study for formulating any strategy for proper utilization of the Kasnau-Matasukh lignites of Rajasthan.

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