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Source rock potential for hydrocarbon generation of Makum coals, Upper Assam, India

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The Makum Coalfield is the most important coalfield in Northeast India as far as coal resources are concerned. In this communication, an attempt has been made to evaluate the possibility of coal as a source material for oil and gas generation. Twenty-five coal samples were collected from different collieries of the Makum Coalfield and have been analysed using Rock-Eval and vitrinite reflectance with coal petrography. It has been observed that the total organic matter content of the Makum Coalfield varies between 59.11 and 84.55% and the hydrogen indices of the studied samples ranges from 150 to 354 mg Hc/g Corg and contain predominantly type II kerogen. Vitrinite reflectance (0.49–0.86 Ro%) studies reflect that the maturity regime of kerogen falls within the oil-generation window. Hydrogen indices and T_{max} reflect that the coals are immature and fall in the zone of 40% type II kerogen. The relationship between atomic H/C and atomic O/C reflects that the coals are rich in vitrinite and are of type III kerogen. The higher hydrogen indices reflect increasing amount of lipid-rich material in the Makum Coalfield, either from cutinite, resinite, exinite (terrestrial macerals) or from marine algal materials. Kerogen is type II or type III, mainly gas-prone and tends to be immature with subordinate oil-generation potential.

Keywords: Kerogen, maturation, oil window, source rock potential.

COAL is understood to have a common association with oil and is also thought to be an important source for the generation of gas and oil. The generation of oil-like substance in coal was established long ago[1,2]. Two main constraints have been found for accepting the coals as a source of oil. During burial, coals cannot generate significant quantities of oil and mostly generate gas; they also lack expulsion capacity.

Therefore, the oil generated within coals cannot be expelled. However, evidence for commercial oil accumulation derived from coal is not sufficient. It is known that some liquid hydrocarbons are generated in coal during catagenesis. There are numerous reports of oil shows, small oil seeps and oil-impregnated sand lenses, etc. being closely associated with coals from all over the world.

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The best known oil-prone sequences have already been identified in Indonesia, Australia, New Zealand and Canada[3]. But the controversy always arises regarding the source of oil generation. A number of workers have tried to solve the controversy regarding coal being oil-prone or gas-prone[4–7]. Further, it was shown that the oil-prone coal or any terrigenous kerogen commonly generates oil at higher temperatures compared to the kerogen that originated in marine and lacustrine environments[8–10].

Maturation or rank of organic matter refers to the physico-chemical stage of coalification which is commonly operated by post-depositional subsidence. Vitrinite reflectance can be successfully applied to assess the rank of coal[11–13]. Vitrinite reflectance is also considered to be the most precise parameter for rank evaluation, because the chemistry of humic material changes with temperature consistently throughout the course of coalification. This can be used for assessing the maturation of the source rock[3].

The Rock-Eval pyrolysis and organic petrography are two vital techniques which can be applied for the rapid assessment of maturation and source characteristics of all types of organic matter.

To find out the source-rock potential of Makum Coalfield for oil and/or gas generation, Rock-Eval pyrolytic analyses have been undertaken. Besides, vitrinite reflectance and maceral composition were also determined. Organic petrography has been used in the source-rock assessment.

The regional geological structure of the Makum Coalfield is represented by a well-defined, roughly NE-SW trending, doubly plunging asymmetric syncline known as the Namdang Syncline (Figure 1)[14]. The syncline is narrow in the Namdang area in the SW and gradually widens out towards NE. The trend of the axis gradually changes to NE-SW eastward and to ENE-WSW further east. The northern limb of the syncline is again folded into an anticline known as Ledo Anticline and the corresponding syncline abuts against the Margherita thrust towards north. Towards south, the southern limb of the syncline is cut-off by the Haflong–Disang thrust. Broadly, the coalfield lies within the belt of Schuppen and is bounded by the Margherita thrust in the north and the Haflong–Disang thrust in the south.

All the coal beds occur in the Oligocene Tikak Parbat Formation, the uppermost member of the Barail Group. The Tikak Parbat Formation is underlain by the Baragolai and Naogaon formations.

A significant source-rock horizon is found in the Barail (Oligocene) Group in the form of carbonaceous shales interbedded with thick coal seams. They have good oil and gas source potential. The combined average thickness of the Barail carbonaceous shale and coal source intervals is of the order of 20–30 m. The Barail sediments were probably deposited in a deltaic environment[15].

The lithologic characteristics of these formations are summarized in Table 1.

The Rock-Eval pyrolysis was carried out for 25 selected coal samples using Rock-Eval-6 apparatus. A weighted sample of 5 mg of crushed coal was placed in the crucible which was inserted into one of the holders of the automatic sampler fitted to the pyrolysis furnace at 300°C for 5 min in the presence of helium. This was followed by programmed pyrolysis at 450–550°C in helium atmosphere. Allowing for the oven to cool, each analysis requires about 30 min.

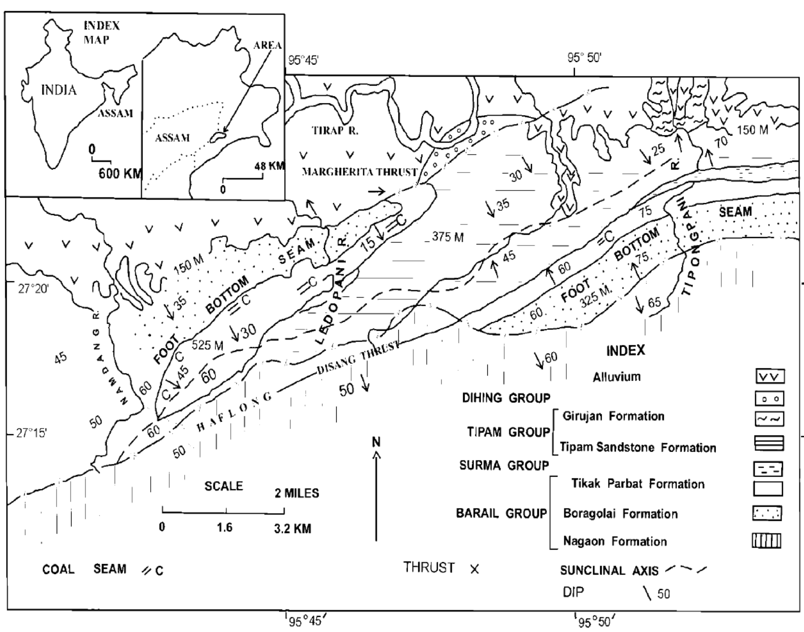

Figure 1. Geological map of the Makum Coalfield (after Goswami[23]).

Table 1. Subdivisions of the Oligocene Barail Group

Formation	Thickness (m)	Description
Tikak Parbat	300–600	Well-bedded and massive, fine-grained sandstones, sandy shales, shales and clays with thick coal beds in the basal part
Baragolai	300	Well-bedded and massive micaceous or ferruginous sandstones alternating with clays, sandy clays and carbonaceous shales and a number of thin coal beds
Naogaon	1100–1700	Thin-bedded, fine-grained quartzitic sandstones with thin partings of splintery shales and sandy shales

Table 2. Indication of source-rock potential based on total organic carbon (TOC) values (after Peters[14])

TOC values (wt%)	Source-rock implications
<0.5	Negligible source capacity
0.5–1.0	Possibility of slight source capacity
1.0–2.0	Possibility of moderate source capacity
>2.0	Possibility of good to excellent source capacity

A flame ionization detector (FID) recorded the organic compounds generated during pyrolysis. The first peak (S_1) gives the amount (in mg) of hydrocarbon that can be thermally distilled from 1 g of coal. The second peak (S_2) gives the amount (in mg) of hydrocarbon that may be generated by pyrolytic degradation of 1 g of coal. A third peak (S_3) marks the amount (in mg) of carbon dioxide generated from 1 g of coal.

The parameters given by this apparatus are: (1) T_{max}: Temperature at which maximum amount of hydrocarbon is generated, which is a measure of the maturity of organic matter. (2) TOC: Total organic carbon (in wt%) present in a coal. (3) Hydrogen index (HI) [$(100 \times S_2)$/TOC]: Parameter used to characterize the origin of the organic material. (4) Oxygen index (OI) [$(100 \times S_3)$/TOC]: Parameter that indirectly correlates the ratio of oxygen to carbon. (5) Productivity index (PI) [$S_1/(S_1 + S_2)$]: Characterizes the evolution level of the organic matter.

For vitrinite reflectance polished blocks from pillar and channel samples were prepared by standard techniques to measure the reflectivity of the coal samples. These polished pellets were studied under reflected light in an oil immersion at 546 nm wavelength light by a microscope photometer (MPV-2). The instrument was calibrated and again it standardized by known reflectance standard of Leukosapphire (Ro = 0.579%). All readings were taken relative to this standard.

Source-rock potential of the analysed samples has been evaluated on the basis of three characteristics, namely quantity, quality and maturity of the organic matter.

The results of the samples with respect to these three characteristics are presented in Table 2.

A commonly accepted minimum total organic carbon (TOC) content for a potential source rock[16] is 0.5%. Rocks containing less than 0.5% TOC are considered to have negligible hydrocarbon source potential. Between 0.5 and 1.0% TOC indicates marginal and more than 1% TOC often has substantial source potential. TOC values between 1 and 2% are associated with depositional environments intermediate between oxidizing and reducing, where preservation of lipid-rich organic matter with source potential for oil can occur. TOC values above 2% often indicate highly reducing environment with excellent source potential. The total organic matter content of the Makum Coalfield varies between 59.11 and 84.55% (Table 3). Such high values are normally indicative of good to excellent source rock potential for hydrocarbon generation (Table 2).

Interpretation of TOC values therefore does not simply focus on the quantity of organic matter present. Many rocks with high TOC values have little oil-source potential because the kerogen they contain may be woody or highly oxidized. Further, TOC values are used as screens to indicate which rocks are of no interest (<0.5%), which might be of slight interest (between 0.5 and 1.0%) and which are definitely worthy of further consideration (>1.0%)[17]. Thus, high TOC values are a necessary but not a sufficient criterion for good source rocks.

HI values and hydrocarbon generation potential, i.e. S_2 of Rock-Eval pyrolysis are indicative of the quality of organic matter. Rocks with S_2 values more than 5 mg/g of rock are considered to be good source rocks. Table 3 shows that all the studied samples have S_2 greater than 5 mg/g. HI values below 150 mg Hc/g Corg indicate the absence of significant amount of oil-generative lipid materials and confirm the kerogen as mainly types III and IV. HI values above 150 mg Hc/g Corg reflect increasing amounts of lipid-rich materials (cutinite, resinite, exinite) or marine algal materials. Samples with HI values between 150 and 300 contain type-III kerogen than type-II and therefore, have marginal to fair potential for liquid hydrocarbons. HI for 16 of the studied samples produced values greater than 300 mg Hc/g Corg and remaining eight samples in the range 150–300 mg Hc/gm Corg (Table 3). The higher HI values, i.e. above 150 mg Hc/g Corg reflect increasing amount of lipid-rich materials in Makum Coalfield either from cutinite, resinite, exinite (terrestrial macerals) or from marine algal material.

Table 3. Source rock and petrographical data of Makum Coalfield

Sample no.	S_1	S_2	S_3	PI	T_{max}	HI	OI	TOC	H/C	Atomic O/C	Atomic Ro%	Vitrinite (%)	Exinite (%)	Inertinite (%)
1	1.08	149.92	1.1	0.01	422	254	25	59.11	0.72	0.08	0.53	77.65	3.74	15.36
2	1.92	184.69	3.05	0.01	429	287	15	64.26	0.72	0.12	0.81	76.09	3.72	18.83
3	3.73	239.67	1.26	0.02	428	346	23	69.34	0.76	0.14	0.79	79.32	2.52	15.52
4	3.53	249.33	2.83	0.01	424	334	14	74.63	0.78	0.16	0.74	81.03	1.82	13.3
5	1.62	220.72	3.11	0.01	425	297	14	74.38	0.76	0.19	0.71	76.25	3.22	18.46
6	2.01	228.62	3.19	0.01	425	293	14	77.99	0.74	0.12	0.67	78.54	1.35	15.76
7	1.23	180.91	3.22	0.01	422	254	15	71.17	0.73	0.11	0.66	78.06	1.17	14.72
8	4.06	267.36	2.32	0.01	429	354	23	75.52	0.76	0.17	0.57	79.24	2.16	12
9	1.65	171.84	4.64	0.01	427	251	27	68.39	0.73	0.09	0.56	82.02	1.7	13.08
10	2.83	252.76	2.38	0.01	422	331	23	76.38	0.75	0.13	0.86	82.62	2.16	11.17
11	3.6	279.48	3.99	0.01	427	331	15	84.55	0.74	0.08	0.66	77.35	1.27	18.08
12	3.49	271.57	2.84	0.01	428	344	14	78.99	0.76	0.14	0.57	78.25	1.52	14.73
13	3.54	261.28	2.48	0.01	425	337	23	77.63	0.74	0.08	0.81	81.76	3.25	8.69
14	3.33	263.75	3.45	0.01	426	343	14	76.8	0.72	0.14	0.58	80.05	2.95	11.4
15	4.09	274.86	4.16	0.01	434	350	15	78.62	0.76	0.14	0.49	78.05	1.28	16.42
16	1.92	259.16	2.86	0.01	431	329	24	78.76	0.73	0.08	0.67	76.5	1.98	18.05
17	1.99	248.56	3.03	0.01	430	303	24	82.01	0.83	0.20	0.69	81.86	1.25	13.73
18	4.19	240.49	1.54	0.02	429	301	12	79.88	0.73	0.10	0.74	77.92	2.18	13.6
19	4.26	238.38	3.64	0.02	421	326	25	73.17	0.74	0.08	0.57	81.54	1.25	14.16
20	1.75	216.87	2.35	0.01	433	284	13	76.35	0.78	0.17	0.79	80.04	2.1	13.91
21	3.2	255.81	1.8	0.01	429	347	22	73.69	0.72	0.09	0.73	78.06	1.57	17.07
22	4.78	262.11	1.85	0.02	437	343	21	76.39	0.66	0.06	0.70	74.43	4.32	18.04
23	2.44	214.04	2.3	0.01	429	297	23	72.12	0.78	0.17	0.74	78.39	1.65	17.08
24	5.02	241.2	1.56	0.02	429	334	22	72.28	0.74	0.08	0.68	79.23	2.12	16.74
Avg.	2.97	236.39	2.71	0.01	427.54	315.42	19.17	74.68	0.74	0.12	0.68	78.93	2.18	15.00
Min.	1.08	149.92	1.1	0.01	421	251	12	59.11	0.66	0.06	0.49	74.43	1.17	8.69
Max.	5.02	279.48	4.64	0.02	437	354	27	84.55	0.83	0.20	0.86	82.62	4.32	18.83

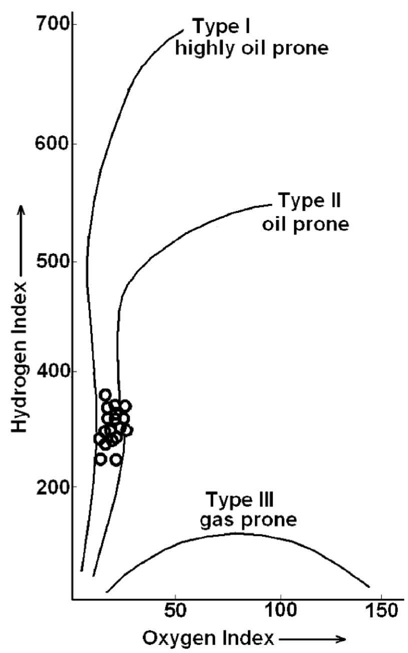

Figure 2. Cross plot of HI vs OI (after Peters[14]).

The most popular method for the characterization of organic matter in terms of kerogen type is that of Van Krevelen method (1961) based on atomic H/C vs O/C ratios. But the commonly used Peters[16] diagram, i.e. the cross plot of HI vs OI (Figure 2) has been used for taking maturity effects into account in the evaluation of pyrolysis data. Figure 2 indicates that the coals of Makum Coalfield are of type-II path line, which is oil-prone. However, Peters[16] has indicated that HI vs OI may misrepresent the type of organic matter for coals and misinterpret the results. Espitalie *et al.*[18–20] demonstrated that oxygen in kerogen is proportional to the liberated carbon dioxide (S_3), and hydrogen content is proportional to the liberated hydrocarbons (S_2) during pyrolysis.

Pyrolysis temperature T_{max} (°C) is an index of maturity because as maturity of kerogen increases, the temperature at which the maximum rate of pyrolysis occurs also increases. In the present studied samples, the PI value ranges between 0.01 and 0.02 and T_{max} value ranges from 421°C to 437°C (Table 3). The cross plot of atomic HI vs T_{max} after Espitalie *et al.*[21] (Figure 3) reflects that the coals of Makum Coalfield lie in the immature zone. Further, Peters[16] has suggested that PI and T_{max} values less than about 0.1 and 435°C respectively, indicate organic matter to be immature, whereas T_{max} greater than 470°C points to the wet gas zone. The plot of atomic HI vs T_{max} (Figure 3) differs from that of HI vs OI (Figure 2) and indicates that the samples of the present study are either immature or in a very early stage of maturation.

However, as indicated by the plot of atomic H/C vs O/C after Tissot *et al.*[22,23] (Figure 4), the organic matter of Makum Coalfield is of type-III (Vitrain) and is gas-prone. It is also evident from the plot of HI vs T_{max} (Figure 5) that majority of the present study samples fall within 40% type-II kerogen limit and tend to be gas-prone with subordinate liquid hydrocarbon generation potential.

Vitrinite reflectance techniques were developed about a half century ago for measuring the rank of coals, in which the vitrinite maceral is usually common. There are, however, many problems with vitrinite reflectance as applied to kerogens. Their application in assessing kerogen maturity may in fact be based on a coincidence that is not always valid[17]. Despite its weakness, vitrinite reflectance is the most popular technique today for estimating kerogen maturity. The vitrinite reflectance values of the present samples ranged from 0.49 to 0.86 Ro% (Table 3). A cross plot between HI vs vitrinite reflectance values of the present samples after Leythaeuser *et al.*[19,24] (Figure 6) shows that kerogen content is of type-II. Waples[17] has suggested that for most kerogens the onset of oil generation is taken to be near 0.6Ro%. The maximum reaches near 0.9Ro% and the end of liquid hydrocarbon generation is thought to be about 1.35Ro%. Therefore, the organic matter in all the studied samples is within the oil window and there is the possibility of hydrocarbon generation from coals.

However, the cross plot between H/C vs Ro% (Figure 7)[25–28] indicates that the coals of the present study show the presence of humic kerogen and are gas-prone.

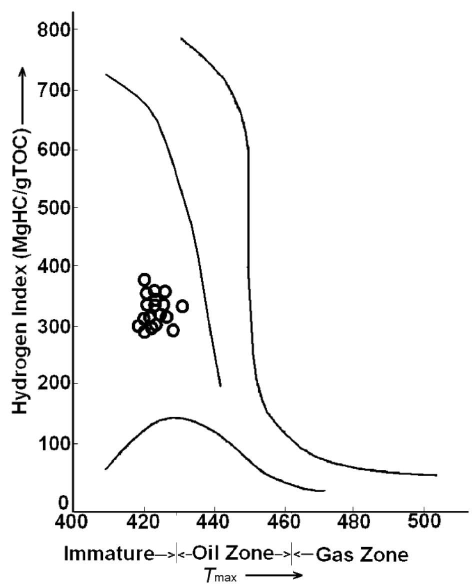

Figure 3. Cross plot of HI vs T_{max} (after Espitalie *et al.*[17]).

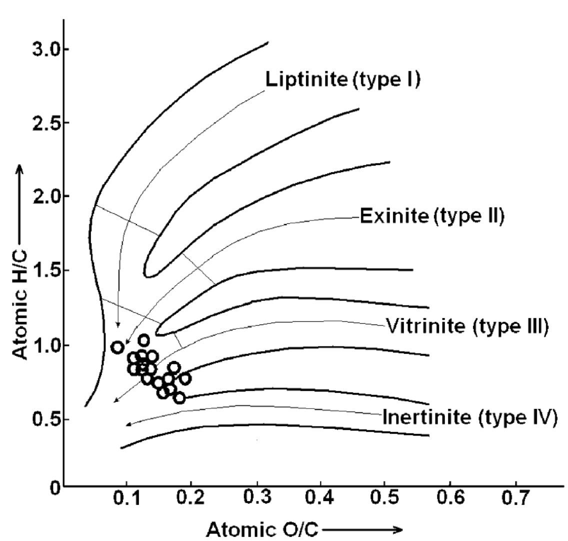

Figure 4. Cross plot of atomic H/C vs atomic O/C (after Tissot *et al.*[18]).

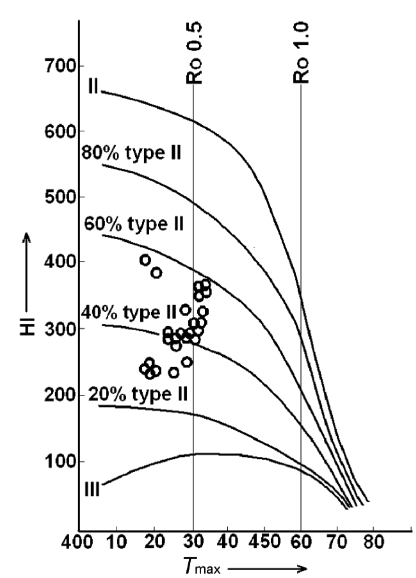

Figure 5. Cross plot of HI vs T_{max}. (after Leythaeuser *et al.*[19]).

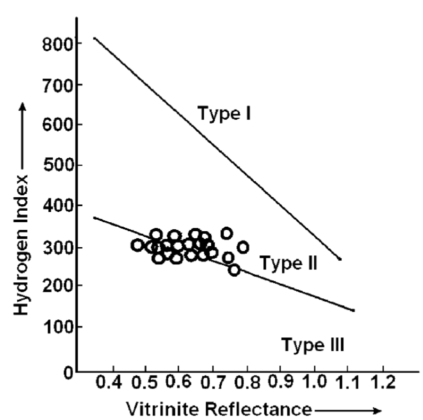

Figure 6. Cross plot of HI vs Ro% (after Leythaeuser *et al.*[19]).

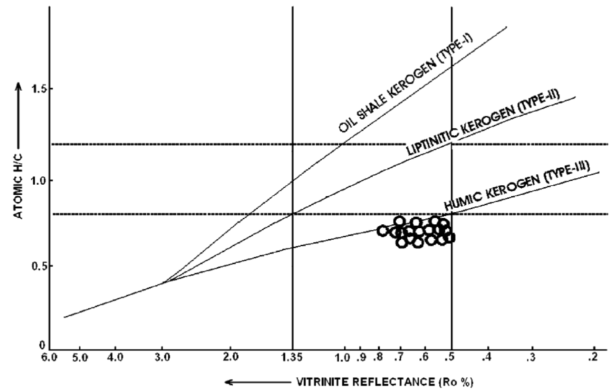

Figure 7. Cross plot of H/C vs Ro% (after Dow[20]).

The importance of resinite/liptinite macerals has been emphasized[23,29–31] for evaluating the source potential of coals. Besides contributing towards oil potential, these macerals also form a network which helps in the migration of hydrocarbons[23,29]. The dominant maceral of Makum coals in average is vitrinite (78.93%). The next in abundance is the inertinite group of macerals, which constitutes 15% of the coal. The amount of exinite (resinite/liptinite, cutinite) varies between 1.17 and 4.32%. From the maceral composition, Makum coals can be classified as gas-prone.

The coals of Makum Coalfield contain relatively high amounts of TOC and high reflectance values indicate predominantly type-II kerogen. The relation between HI and vitrinite reflectance indicate the coals are of type-II kerogen, and that between H/C and Ro% show that the coals are of type-II humic kerogen. However, the plot between HI and $T_{\max}$ reflects that the coals are immature and fall in the zone of 40% type-II kerogen. The relation between atomic H/C and atomic O/C reflects that the coals are rich in vitrinite and are of type-III kerogen. The above studies indicate that the coals of Makum Coalfield are either immature or in a very early stage of maturation. The type of kerogen is II to III, mainly gas-prone with subordinate oil-generation potential.

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Intraspecific variation and interrelationships between morphology, nutritional content and enzymatic activity of *Jatropha curcas* L.

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The study was conducted to assess intra-specific variation and interrelations among morphological, nutritional and biochemical traits in 27 accessions of *Jatropha curcas* L. Principal component analysis (PCA) explained 58% of the total variation in the measured traits and revealed that there was negative correlation between four morphological traits and all nutritional compounds such as crude protein, neutral detergent fibre, acidic detergent fibre, lignin, hemicellulose and cellulose. Larger plants tended to contain lower concentration of protein and other nutritional compounds. Female flower/inflorescence showed maximum variation, while the difference for the number of male flowers/inflorescence was narrow. This might be explained by the difference in intracellular and extracellular enzymatic activity at different development stages in *J. curcas* L. A strong correlation between plant height and branch length, number of branches and collar diameter was observed, which can help in the selection of superior genotypes of this species. The highest genetic variation was noticed for polyphenol oxidase analysis. PCA analysis showed that peroxidase activity was associated with cellulase, acid detergent fibre and protein content, whereas polyphenol activity was associated with neutral detergent fibre and lignin content.

Keywords: Enzymatic activity, intraspecific variation, *Jatropha curcas* L., nutrient content.

INDIA produces only 30% of its annual crude oil requirement of 111 mt and hence depends on imported crude oil to the tune of Rs 102,500 crores for meeting the requirements, resulting in a huge burden on the country's economy. This together with the rapid depletion of fossil-fuel reserves, high prices and associated environmental degradation with their use has prompted a search for alternative fuels in the world. In this scenario, the production and use of biofuels is one of the ways to reduce our dependence on fossil fuel and protect the environment. Biodiesel produced from edible and non-edible oils of plant origin and animal fat provide alternative sources of biofuel. As India is not self-sufficient in edible-oil requirement, focus is drawn on the production of biodiesel from non-edible oil-seeds. According to an estimate, even 5% replacement of fossil fuel by biodiesel will help save foreign exchange of over Rs 4000 crores annually[1]. Many non-edible tree-borne oil-seed species such as Jatropha, Karanj, Pilu, Mahua, Sal and Cheura, widely found in India, are suitable for biodiesel production. Among these, *Jatropha curcas* is recognized as most potential species for biodiesel production, since the seeds contain high oil content (30–38%) and could be grown under different land-use situations[2]. It can be easily propagated by seeds or cuttings and starts bearing within 2 to 3 years. Also, it can be commercially exploited in 4–5 years and lasts for about 50 years[3]. The species is not grazed by cattle and withstands extreme drought conditions. Out of 146 mha of wasteland existing in India[4], plantation of *Jatropha* will help greening a large portion of it. As *Jatropha* is fast-growing and produces huge quantity of vegetation, it will add substantial quantity of organic matter to the soil through litter fall and fine root turnover. The increased organic matter will improve the physico-chemical properties of degraded lands. Considering its potential as a bio-energy crop, efforts are being made worldwide to enhance the genetic potential of *Jatropha* for economical biodiesel production. The availability of genetic variability and the knowledge of inter-relationships between different morphological and economic traits such as seed yield and seed biochemical traits are a prerequisite for systematic *Jatropha* genetic improvement. Therefore, better knowledge of physiology and biochemistry of *J. curcas* is

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