Evaluation of Shale Gas Reservoir in Barakar and Barren Measures Formations of North and South Karanpura Coalfields, Jharkhand

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Abstract: India recognizes the strategic importance for developing shale gas resources like other countries in the world. Shale gas reservoirs are known to be difficult for extracting gas in comparison to conventional reservoirs. Recently, due to high prices of gas, rising demand and enhancement in recovery technologies has attracted the Indian energy industries to explore the shale gas resource. Coal and lignite are the prime source of energy in India and these resources are well explored, while shale is ignored, despite it being associated with coal and lignite bearing formations. The paper presents reservoir characteristics of shale horizons in Barren Measures and Barakar formations of north and south Karanpura coalfields. Shale core samples were collected from exploratory boreholes in air tight canisters. In-situ gas content and adsorption capacities ascertained to be $0.51-1.69 \text{ m}^3/\text{t}$ and $3.90-5.82 \text{ m}^3/\text{t}$ trespectively. Desorbed gas derived from canisters contains CH₄, C₂H₆, C₃H₈, CO₂, N₂ and O₂ and varies from 76.19-82.63, 0.38-0.76, 0.10-0.50, 8.65-12.34, 9.89-19.34 and 0.56-2.24 vol. % respectively. The permeability and porosity determined under reservoir simulated confining pressure is varying from 0.41-0.75 mD and 0.89-2.28 % respectively. The plots of Rock Eval S₂vs TOC and HI against Calc. VRo% indicates that all shale samples belong to Type III kerogen, which is prone to generate gas. It is evaluated that insitu gas content, sorption capacity, saturation level and low permeability of shale beds are critical parameters for development of shale gas resource in the studied area.

Keywords: Shale gas;reservoir potentiality; Barakar Formation; porosity; permeability; North and South Karanpura Coalfield, Jharkhand

INTRODUCTION

Gas from shale reservoirs has rapidly emerged as an economic resource due to advances in horizontal drilling and hydro-fracturing technologies, which aid its production from such reservoirs which have intrinsically low permeability. Shale is a fine-grained, typically less than 4µm (0.004mm) clastic sedimentary rock composed mostly of clay (>65%) and is mixed with flakes of quartz, mica and micro fragments of other minerals. Shale is characterized by breaks along thin laminae or parallel layering or bedding less than one centimeter in thickness, called fissility (Blatt and Robert, 1996). Geologically, shale is recognized as a good cap rock for conventional reservoirs because of its very low permeability. Shale normally has higher porosities than sand, but most of it is micro and disconnected porosity. Shale has extremely low permeability compared to most of the conventional reservoirs like sandstone, limestone or dolomite.

Gas is stored in shales in three different states: *adsorbed gas* that is attached to organic matter or to clays,

free gas that is held within the micro-pores and microfractures in the rock or in spaces created by the rock cracking and solution gas that is held in a dissolved state in the liquid phase occupying the pores. The amount of adsorbed gases usually increases like CH4, C2H6 and C3H8 etc or non hydrocarbon gases such as CO2, NO2, CO etc which can be adsorbed in natural state with an increase in organic matter or surface area of organic matter and clays (Daniel and Bustin, 2009). As adsorbed gases which are detained within in micro-pores and/or fractures due to molecular forces, it normally requires further excitation or pressure to release it while free gas which resides in pores and/or fractures is easily released on opening of the pores or fractures. This is because the free gas resides in fractures and pores and get out easily relative to adsorbed gas. Higher free-gas content in unconventional shale gas wells generally results in higher initial rates of production.

The aim of the present study is to determine the gas desorption and adsorption potential, porosities and

permabilities of the shales and derive their gas production potential.

SHALE GAS IN INDIA

A decade ago natural gas from shale was just becoming significant but hardly anyone fathomed its tremendous potential or its parabolic growth. India energy mission has given a prime importance to explore shale gas potential in the country (Rao, 2013). Initiative has been undertaken by the Central Institute of Mining and Fuel Research (CIMFR) and Oil and Natural Gas Corporation (ONGC) for shale gas exploration in the Damodar basin. The preliminary results are encouraging and need detailed reservoir evaluation of shale gas for commercial viability of this basin (Mendhe, 2014). Geologically, India possesses large shale deposits in Gondwana basins and across the Gangetic plain, Rajasthan, Gujarat, Assam-Arakan and other coastal areas, but none of these have been explored for gas (MoPNG, 2013). The longterm success of shale gas in India depends on two factors, namely, technology and constructive policy. Shale gas activities will promote, only when cutting-edge technology is made available ensuring economical viability. The government of India approved a policy to allow state-owned companies to start exploration and exploitation of shale gas in September 2013, to develop alternatives energy resources aiming in reduction of expensive oil imports. It is emphasized that of about 356 blocks owned by ONGC and Oil India Ltd, India's in which 176 maybe explored for shale gas resources (MoPNG, 2013). India could be sitting on as much as 96 trillion cubic feet (tcf) of recoverable shale gas reserves, equivalent to about 26 years of its gas demand, according to the US Energy Information Administration (EIA, 2011).

WORLD STATUS IN SHALE GAS

Worldwide, only the USA has a full grip on shale gas exploration and production from different basins due to advance technologies. Annual production from shale-gas reservoirs in the USA is approximately 1.0 Tscf and comes from more than 40,000 shale gas wells completed in five primary basins. Shale gas continues to be one of the hottest plays in the USA and drilling is expanding rapidly, especially in the south-central USA (the Barnett shale and its equivalents), the Appalachian basin (Marcellus Formation), and numerous Rocky mountain basins. No commercial shale-gas projects currently exist outside USA, but work continues to identify both new shale-gas reservoirs and to add incremental shale-gas production in existing reservoirs. Given that worldwide shale-gas resources are estimated to exceed 16,000 Tscf, it is clear that tremendous potential exists for future growth (Kawata and Fujita 2001). Recently, Canada, UK, Australlia, China and Russia have taken interest in shale gas exploration.

Technology drives the exploration and production of shale gas, because advanced and innovative technologies are needed to access low-permeability reservoirs. The advancement in horizontal drilling to maximize the drainage area of a well, and rapid increases in natural gas prices in the last several years resulted in the significant development of shale gas reservoir. By drilling number of horizontal wells from a single drilling pad, to different sub-surface locales and directions, the surface footprint is reduced. The objective of not drilling vertical wells is that they simply are unable to give an economic production rate whereas on the other hand horizontal wells and multiple hydro-fracturing help to achieve the economic gas production. Although shale gas has been produced for more than 100 years in the Appalachian basin and the Illinois basin of the United States, the wells were often marginally economical. While unconventional gas sources like shale gas reserves are plentiful, cost to produce is more than the conventional gas production. The shale gas current cost has been estimated to be between \$3 to 4 mmbtu (Million Metric British Thermal Units) in the world market (EIA, 2015).

STUDY AREA – DAMODAR BASIN

In this investigation, two adjacent Gondwana coalfields viz. north and south Karanpura are studied for shale reservoir characteristics to better understand the shale gas resource potential. The geological details of aforesaid two coalfields are discussed as below.

North and South Karanpura

The north Karanpura coalfield covering an area of about 1230 sq km lies between latitude 23°38'- 23°56' N and longitudes 84°46'- 85°23' and forms the westenmost part of upper Damodar valley coalfields. The major portion of the coalfield lies in Hazaribag district, while the western and southern fringes occur in Palamau district of Jharkhand (GSI, 1977 and CMPDIL, 1993). The coalfield is a saucershaped basin having a maximum length of 64 km in the eastwest direction and a width of about 36 km in the north-south direction (Figure 1).

The south Karanpura coalfield covering an area of about 175 sq km lies between latitude 23°38' - 23°43' N and longitudes 85°07'- 85°28' E. Situated mostly in Ranchi district and a small portion in Hazaribagh district of



Fig.1. Location of shale core samples in (a) North Karanpura and(b) South Karanpura coalfields (modified after CMPDIL, 1993).

Jharkhand. The field forms a part of the upper Damodar valley coalfields and is a major contributor of coal in the Jharkhand state (GSI, 1977 and CMPDIL, 1993). The south Karanpura coalfield is a narrow semi-elliptical basin having a maximum east-west extension of about 37 km, while its width is less than 9 km in the north-south direction,

(Fig. 1). The thickness of this sedimentary deposit is about 1500 meters, containing upper Gondwana lithostratigraphic unit like Mahadeva and lower Gondwana repersented by Panchet, Damuda and Talcher.

Shale Beds Occurrence

A thick sedimentary succession ranging from Carboniferous to Cretaceous age exists in north and south Karanpura coalfields of Damodar valley including shale beds of varying thickness laterally. Apparently there are no published data or information dealing with the shale gas potential and properties of the shales present in the area.

Karharbari, Barakar, Barren Measures and Panchet Formations having thick shale beds with cumulative thickness of about 500 – 800 in the north Karanpura coalfield. The south Karanpura coalfield has sediments of Karharbari, Barakar and Barren Measures containing shale beds of about 300-500 m thick.

EXPERIMENTS AND RESULTS

Shale Core Sampling

A total of 6 shale core samples were collected out of which 3 samples came from two boreholes BHN1 and BHN2 in north Karanpura (Barren Measures - NK-S1, NK-S2 and Barakar Formation – NK-S3) and 3 samples from two boreholes BHS1 and BHS2 in south Karanpura (Barren



Generalised litho-stratigraphic succession of North and South Karanpura coalfields (after GSI, 1977 and CMPDIL, 1993).

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Sample No.	Depth of Sample (m)	Surface features and visible minerals	Physical properties
NK-S1	511	Irregular fracture, muscovite dominantly present throughout the sample in very homogeneous manner in the form of rounded as flecks.	Grey in colour, dull lustre, dirty white streak, non-banded, very fine grain with clay, >2.5 hardness, uneven to sub conchoidal fracture and about 2.4 sp. gravity.
NK-S2	621	Irregular fracture filled with mud, muscovite dominantly present through-out the sample with homogeneous manner in the form of rounded flecks.	Grayish-black colour, dull lustre, dirty white streak, non-banded, fine grains with clay, >2.5 hardness, uneven fracture and about 2.3 sp. gravity.
NK-S3	791	No fractures observed in the samples, muscovite mica is insignificant throughout the sample.	Black in colour, dull lustre, dirty white streak, non-banded, very fine grain size, hardness >2.5, uneven surfaces and 1.9 sp. gravity.
SK-S1	828	Surfaces are even to uneven and broken along the bedding plane and has fossil imprints. It also contains mud fillings and muscovite flecks present throughout the sample.	Dark grey to black in colour, dull to vitreous lustre, dirty white streak, non-banded, fine to very fine grain size, >2.5 hardness, uneven fracture and 2.7 sp. gravity.
SK-S2	682	Irregular fractures with mud/clay fillings, muscovite present in heterogeneous manner in the form of very small flecks.	Dull black in colour, dull lustre, black streak, non-banded, very fine grains with clay, >2.5 hardness, even surfaces and 1.7 sp. gravity.
SK-S3	838	No fractures, muscovite dominantly present throughout the sample in very homogeneous manner in the form of rounded flecks.	Dark grey in colour, dull to vitreous lustre, grayish white streak, non-banded, very fine grain size, >2.5 hardness and uneven fracture surfaces and 1.8 sp. gravity.

Table 1. Megascopic properties of shale core samples.

Measures - SK-S1 and Barakar Formation – SK-S2, SK-S3) coalfields during exploratory drilling for coalbed methane. Location of the boreholes are marked and shown in Figure 1. Shale core samples were collected in air tight cylindrical canisters. Megascopic analysis of shale core samples was carried out before crushing the samples for different analyses. The detailed descriptions and photographs of investigated shale core samples are presented in Table 1 and Fig.2 respectively.

In-Situ Gas Content by Direct Method

The Direct method for determination of *in-situ* gas content of shale core samples has been followed in the

investigation. The method which was originally suggested by Bertard *et al.*, 1970 for determination of gas content of coal seam being worked underground has been modified by several authors (Diamond and Levine, 1981; Kissel et al., 1973; McCulloch et al., 1975; Feng et al., 1984) for its application in coal core samples collected from exploratory boreholes drilled for estimation of coal resources. As there is no separate and established method for *in-situ* gas content determination of shale core samples, the direct method used for *in-situ* gas content of coal is applied in this investigation to estimate the *in-situ* gas content of the shale core samples.





Fig.2. Megascopic photographs of shale core samples obtained from North and South Karanpura coalfields (Sample NK –S1, SK-S2 and SK-S3).

consists of specially fabricated temperature controlled gas tight cylindrical stainless steel canisters of $2\frac{1}{2} \times 18\frac{1}{2}$ and $4\frac{1}{2} \times 18\frac{1}{2}$ size fitted with a pressure gauge and a needle valve and a manometer type of apparatus for measuring gas volume. After desorption measurements, a known quantity of the sample was crushed to fine powder below 200 mesh BSS size in a hermetically sealed grinding mill, purged with inert gas argon to avoid absorption of released gas by air or oxygen. The volume of released residual gas after crushing was measured volumetrically (Mendhe et al., 2010).

There are mainly three steps involved in measurement of in-situ gas content of shale core samples retrieved from exploratory wells *viz*. estimation of lost gas (Q_1) , measurement of desorbed gas (Q_2) and evaluation of residual gas (Q_3) . The *in-situ* gas content determined by direct method is given in Table 2.

The total volume of gas obtained by the addition of Q_1 , Q_2 and Q_3 gives the *in-situ* gas content (Q) of the sample when divided by the total weight of the sample.

$$Q = \frac{Q_1 + Q_2 + Q_3}{\text{Wt. of the sample}}$$
(1)

Shale samples are analysed for moisture, inorganic, volatile matter and fixed carbon content (Table 2) by following standard laboratory procedure as laid down by Bureau of Indian Standard (BIS: 1350) to correlate the shale samples on dry ash free basis.

High Pressure Adsorption Isotherm

Determination of adsorption isotherm is a three step process namely sample preparation, void volume estimation after putting the samples in the sample cells and adsorption capacity determination. The shale samples were crushed in order to obtain -0.630mm +0.400mm size. Equilibrated moisture of shale samples were prepared following the procedure (IS: 1350; Part-I and ASTM Standards, 1997), in which the samples were weighed and dried at 50°C and pre-wetted with deionized water and known weight of sample is placed in an evacuated desiccator containing water vapour in equilibrium with saturated solution of K_2SO_4 at 30°C and periodically reweighed and placed in an evacuated desiccator until a change in weight does not exceed 0.001g.

The dead volume of the apparatus is determined by evacuating and then filling the reference cell with helium and allowing it to equilibrate. Helium is then admitted into the sample cell and dead volume may be calculated by drop in pressure. After the determination of dead volume, the apparatus is again evacuated for determination of the adsorption isotherm. The adsorbent is added to the reference cell, allowed to equilibrate and then admitted into the sample cell. The amount of gas adsorbed is determined from drop in pressure. The process is repeated at increasing pressure until the highest pressure is reached (Crosdale, 2000). The result on adsorption capacity of shale samples is presented in Table 2.

The following Langmuir adsorption isotherm model is used for plotting and estimation of Langmuir coefficients:

$$V_a = V_L \ \frac{P}{(P_L + P)} \tag{2}$$

Where, V_a – Adsorbed volume, V_L – Langmuir volume, P_L – Langmuir pressure and P – equilibrium pressure

Desorbed Gas Quality

The desorbed gas progressively replaced air originally present in the void volume of the canisters and was analyzed for the composition of desorbed gas for each canister before they were opened for residual gas measurement. Both the detectors flame ionization detector (FID) and thermal conducting detector (TCD) of gas chromatography at CIMFR, Dhanbad have been used for determination of the concentration of methane, nitrogen and carbon dioxide. The oxygen concentration estimated by difference. To know the hydrocarbon percentage and its distribution with time, the FID detectors were used to analyze higher hydrocarbon concentration in desorbed gas as per the procedure given

Fable 2	. In-situ	gas	content	of	shale	core	samples	s by	direct	meth	lod	
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Sample No.	Gas content (m ³ /t)	Moist (wt.%)	Ash (wt.%)	Volatile matter (wt. %)	Fixed Carbon (wt.%)	Gas content (daf)(m ³ /t)	Reservoir temperature (°C)	Max.pressure applied (kPa)	V_L (m ³ /t)	P _L (kPa)
NK-S1	1.24	1.45	78.36	12.64	6.31	6.14	45	8628	4.70	5218
NK-S2	1.11	1.63	74.92	15.86	6.48	4.73	48	8639	4.50	5311
NK-S3	1.69	1.13	71.27	14.21	11.70	6.12	54	8608	4.80	5223
SK-S1	1.08	0.44	89.24	7.64	1.60	10.47	55	8451	4.30	5717
SK-S2	0.58	0.56	69.87	13.46	15.53	1.96	50	8523	5.40	5276
SK-S3	0.51	0.63	72.20	18.39	8.27	1.88	55	8465	5.20	5506

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Table 3. Gas composition for quality by chromatography

Sample No.	Sample Com- No. bustible		N_2	O_2	Hydrocarbons in combustible gas				
	gas			(1.0/)	CH_4	C_2H_6	C_3H_8		
				(vol. %)					
NK-S1	75.38	8.65	15.36	0.61	76.19	0.67	0.10		
NK-S2	74.19	9.29	16.33	0.19	80.32	0.45	0.30		
NK-S3	78.21	10.43	9.89	1.47	78.97	0.38	0.20		
SK-S1	69.92	8.96	19.34	1.78	79.06	0.76	0.40		
SK-S2	76.77	11.22	11.45	0.56	76.47	0.76	0.30		
SK-S3	72.34	12.34	13.08	2.24	82.63	0.59	0.20		

by Chemito, 2004. The results on desorbed gas composition along with detailed hydrocarbons in total combustible gas are presented in Table 3.

Porosity and Permeability Under Confining Pressure

Shale cores of original diameter of 101.6mm were reduced to 48 mm diameter core by re-coring in the laboratory. The core plugs used for measurement were perpendicular to the bedding. The length of re-cored shale sample are around 152.4mm in length, the ends of which were trimmed and polished. Placed the core into core holder and allowed it to attain desired confining pressure. Helium gas was injected through the core sample via a pressurereducing gas regulator and a metering valve. A core holder and confining-pressure pump were used to apply uniform confining (simulated overburden) pressure to the sample. A gauge-pressure transducer measures inlet pressure to the core sample. The pressure drop across the core sample was measured with one of three calibrated mass flow meters which had voltage output signals. A thermocouple measures the flowing temperature (ambient temperature) of the gas. The computer data acquisition system records the differential pressure, gas flow rate, gas viscosity, core dimensions, and temperatures. Darcy's equation was applied to calculate the gas permeability. It takes the measurements of the core diameter and length, pressure drop across the core, the gas flow rate at that pressure drop and the flowing gas temperature into consideration for calculation (Temco, 2003).

$$K_{a} = \frac{2\mu Q_{b} P_{b} L T_{act}}{A(P_{1}^{2} - P_{2}^{2}) T_{ref}}.1000$$
 (3)

Where, $K_a = gas$ permeability (mD), $\mu = gas$ viscosity (centipoise, cp), $Q_b = mass$ flow rate of gas (cm³/sec), $P_b =$ standard reference pressure (atm.), L = core length of sample (cm), A = cross sectional area of core sample (cm²), $P_1 =$ upstream pressure (atm), $P_2 =$ downstream pressure (atm), $T_{act} =$ actual flowing temperature of gas (K) and T_{ref} = reference temperature of mass flowmeter (K). Porosity measurement was carried using pycnometer containing reference cell and the same core holder used for permeability. The prepared shale core sample was placed in the core holder, the reference chambers and transducers plumbing (both of known volume) were pressurized to approximately 100 psia (0.69 Mpa, a absolute). Opened the core holder inlet valve and released some amount of gas into the core and core holder plumbing and allowed it to settle. The equilibrium pressure was measured. *Boyle's Law* is used to calculate the pore volume of the sample. Porosity is calculated by dividing the pore volume by the bulk volume (Temco, 2003).

$$V_{pore} = \frac{(V_{tube} + V_{ref})(P_{HI} - P_{LO})}{P_{EQ} - P_{LO}} - V_{dead}$$
(4)

Where, V_{tube} = volume among the tubing and pressure transducer, V_{ref} = volume of reference chamber, P_{HI} = initial high pressure in pressure transducer, P_{LO} = initial low pressure in core sample and dead volume, P_{EQ} = Equilibrium pressure after opening reference chamber and V_{dead} = dead volume in inlet and outlet plumbing and distribution plugs of core holder. To obtain porosity following equation can be used:

Porosity,
$$\varphi = \frac{V_{pore}}{V_{bulk}}$$
. 100% (5)

Where, V_{pore} = volume of pores, V_{bulk} = volume of shale core sample.

The results of measurement of porosity and permeability under reservoir simulated confining pressure are given in Table 4.

TOC and Rock Eval Pyrolysis

Shale samples were ground and passed through 60 mesh (250 micron) sieve for TOC and Rock-Eval pyrolysis analysis. The samples were treated with conc. HCL to remove carbonates and total organic carbon (TOC) is

 Table 4. Porosity and permeability of shale core samples under reservoir simulated confining pressure.

Sample No.	Depth	Helium Inlet Pressure (psi)	Reservoir Simulated Confining Pressure (psi)	Permeability (mD)	Porosity (%)
NK-S1	511	693.05	728.93	0.68	1.13
NK-S2	621	853.82	884.36	0.75	1.58
NK-S3	791	1084.35	1126.08	0.57	2.28
SK-S1	828	1152.72	1178.95	0.63	0.98
SK-S2	682	942.96	970.14	0.54	0.89
SK-S3	838	1152.11	1191.31	0.41	1.22

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			1	able 5. I	COCK-EVa	i pyrofysis d	lata of samples				
Sample No.	S ₁ (mg HC/g)	S ₂ (mg HC/g)	S_3 (mg CO ₂ /g)	PI	T _{max} °C	TOC (wt.%)	HI (mg HC/g TOC)	OI (mg CO ₂ /g TOC)	S_2/S_3	S ₁ /TOC(mg HC/g TOC)	Calc. VRo%
NK-S1	0.12	3.45	0.11	0.03	442	2.27	152	5	31	0.05	0.80
NK-S2	0.19	5.63	0.07	0.03	445	3.22	175	2	80	0.06	0.85
NK-S3	0.26	18.35	0.19	0.01	435	6.89	266	3	97	0.04	0.67
SK-S1	0.09	16.75	1.42	0.03	426	1.11	248	128	2	0.08	0.51
SK-S2	0.38	34.21	3.18	0.01	428	12.34	277	26	11	0.03	0.54
SK-S3	0.31	9.48	1.75	0.03	434	6.89	138	25	5	0.04	0.65

 Table 5. Rock-Eval pyrolysis data of samples

measured on carbonate free samples.Rock-Eval pyrolysis were carried out, according to the methods described by various authors (e.g. Barker, 1974; Claypool and Reed, 1976; Espitalie et al., 1977 and 1984; Clementz et al., 1979; Larter and Douglas, 1982; Horsfield, 1985; Peters and Simoneit, 1982; Peters, 1986) to obtained data on the maturity, maturity and type of the source rocks. The parameters obtained includes total organic carbon (TOC) content, S1 (hydrocarbons released at temperatures of about 300°C), S₂ (hydrocarbons generated by pyrolytic degradation of the organic matter in the rock samples at temperature of 300-550°C), S₃ (CO₂ generated from the kerogen during thermal cracking of the kerogen) and T_{max} (temperature at which the maximum amount of hydrocarbons are generated, which is a measure of thermal maturity of the organic matter). Other parameters calculated from the pyrolysis data are hydrogen index (HI= S₂*100/TOC), oxygen index (OI = S_3 *100/TOC), production index (PI= S_1 / (S_1+S_2)) and calculated vitrinite reflectance (Calc. VRo%=0.0180*T_{max}-7.16) (Ekweozor and Gormly, 1983; Jarvie et al., 2005; Jude 2011). The results of Rock Eval pyrolysis is given in Table 5.

DISCUSSION

Six shale core samples were collected from north and south Karanpura coalfields from different shale beds laterally varying with depth and thickness. The megascopic properties *viz*. depth of occurrence, surface features and visible minerals and physical properties of shale core samples are given in Table 1. The depth of the shale core samples in north and south Karanpura is varying from 511 to 791 m and 682 to 838 m respectively. The studied shale samples are grey to black in colour, having irregular fractures filled with mud, dominance of muscovite flecks and rich inorganic content.

In-situ gas content measured by direct method is varying from 0.51 to 1.69 m³/t on received basis and 1.88 to 10.47 (m³/t) on dry ash free basis (Table 2). The moisture, ash and volatile matter varies from 0.44 to 1.63, 69.87 to 89.24 and

7.64 to 18.39 wt. % respectively. The moisture content decrease with depth is shown in Fig.3. It is inferred that due to overburden pressure shale occurring at greater depth have liberated most of the moisture content. The adsorption capacity at maximum pressure applied is observed in the range of 2.70 to 3.90 m^3 /t on as received basis.

The maximum sorption capacity of shale core samples called Langmuir volume (V_1) and Langmuir pressure (P_1) are determined in the range of 4.30 to 5.40 m^3/t and 5218 to 5717 kPa respectively. The in-situ gas content compared with maximum sorption capacities (V_1) of shale samples revealed that shale beds in the study area are under to moderately saturate. Relationship between Langmuir volume and gas content is given in Fig.4. The inverse relationship between gas content and Langmuir volume emphasized that the gas trapped in the pores has similar properties to the gas which is adsorbed. Free gas trapped in the pores will be independent of the surrounding material whether clay or organic, its entrapment is more related to the pore being isolated/sealed than the surroundings. Desorbed gas composition through gas chromatography viz. combustible gas, CO₂, N₂ and O₂ varies in the range of 69.92 to 76.77, 8.65 to 12.34, 9.89 to 19.34 and 0.19 to 2.24 Vol. % respectively. The distribution of hydrocarbons in total combustible gas viz. CH₄, C₂H₆, C₃H₈ is varying from 76.19 to 82.93, 0.38 to 0.76 and 0.10 to 0.40 % respectively



Fig.3. Moisture content with depth of shale sample.



Fig.4. Gas content vs Langmuir volume.

(Table 3). The concentration of methane in desorbed gas increases with depth as shown in Fig.5, indicating more methane sorption in deeper shale beds, because of higher confining pressure. The occurrence of CO_2 with depth is shown in Fig.6, which indicates that CO_2 concentration increases with depth pointing towards the availability of high temperature gradient at greater depth, which is responsible for generation of CO_2 (Mendhe, 2014). The concentration of O_2 gas increases with depth as shown in Fig.7. The O_2 gas formed during the disintegration of organic matter is also stored in shale because of availability of pore space and significant confining pressure.

The distribution of desorbed combustible and noncombustible gases from the shale samples are plotted in ternary diagram (Figs. 8 and 9). It reveals that combustible gas concentration dominate over the other non-hydrocarbons (Fig.8), while non-hydrocarbons have shown variations in their occurrence (Fig. 9). It can be concluded that desorbed gas from shale is dominated by thermal genes such as thermal degradation of kerogen and thermal cracking of volatile matter (Jarvie et al., 2007).

The gas content v/s porosity and permeability plot (Fig.10) reveals that the with increasing total porosity (pore and micro-fracture) gas content increases. At the outset permeability has a poor correlation with gas content due to critical diffusion mechanism between pores and paths used for migration of gas. The plot of combustible gas with porosity and permeability (Fig.11) shows that combustible gas increases with increasing porosity. It may be observed that combustible gas containing hydrocarbon have affinity towards pores, on the other hand permeability do not have any relation with combustible gas, because of decisive diffusion of gas from pores.

The porosity and permeability measured under



Fig.5. Methane concentration with depth.



Fig.6. Carbon dioxide concentration with depth.



Fig.7. Oxygen concentration with depth.

reservoir simulated confining pressure are observed to be in the range of 0.89 to 2.28% and 0.41 to 0.75 mD respectively (Table 4). Effect of depth and confining pressure on permeability and porosity is shown in Fig.12, which indicates that shale pores, cleats and fractures do not



Fig.8. Ternary diagram of N₂, CO₂ and combustible gas.



Fig.9. Non-hydrocabon in desorbed gas.

have any impact of increasing confining pressure, may be due to higher inorganic matter content, which has greater strength.

The Rock Eval and associated analysis (Table 5) reveal that the TOC content (1.11 to 12.34 wt.%), T_{max} (426 to 445 °C), HI (138 to 277 mg HC/g), OI (2.0 to 128.0 mg CO₂/g), PI (0.01 to 0.03) and Calculated VRo% (0.51 to 0.85%). The plots of Rock Eval S₂vs TOC (Figure 13) is useful to compare the gas generative potential of source rocks (Langford and Blanc-Valleron, 1990; Peters, 1986). The plot of S₂ against TOC for immature to marginally mature samples is used to determine the proportion of the





Fig.10. Gas content with porosity and permeability.



Fig.11. Combustible gas vs porosity and permeability.



Fig.12. Porosity and permeability with depth.

inert carbon in the TOC measured by pyrolysis (Conford et al., 1998). During pyrolysis, only the labile kerogen will generate hydrocarbons to be recorded in S2 peak. It is found that all shale samples belong to Type III kerogen, which is known to be gas prone. The T_{max} values of studied samples indicate immature to marginally mature source rock. The HI values of the studied samples vary between 138 to 277 mg HC/g rock and indicate input of dominantly type III organic materials. Type I kerogen (oil prone) commonly possesses HI values of more than 600 mg HC/g TOC while type II (oil prone) possess values greater than 300 but lower than 600mg HC/g TOC (Hakimi et al., 2012a,b, 2013;



Figs.13-16. Plots of Rock-Eval (13) S₂ vs. TOC. (14) HI vs. OI. (15) HI vs. T_{max} and (16) HI vs. Calc. VRo%.

Nicholas et al., 2004; Peters and Cassa, 1994). A bivariate plot of HI vs. OI (Pseudo-Van Krevelen diagram) is also commonly used to assess the kerogen type (Fig.14). TOC and kerogen type are used to evaluate the hydrocarbon source rock potential based on data given in Table 5 (Fig.14; following Espitalie et al., 1985 and Kotarba et al., 2002) and HI vs T_{max} (Fig.15; modified after Varma et al., 2014b), all indicate input of dominantly type III organic materials. However, all samples have very low OI values (except sample No. SK-S1) corresponding to the input of dominantly type II-III admixed organic materials. Similar results were observed for coals (Rice et al., 1989; Teerman and Hwang, 1991; and Kotarba et al., 2002), however, such observations are also noted for different types of shale (Varma et al., 2014a, 2015a and b). Such OI values may be attributed to the presence of segregated stable oxygen content which is not cracked at relatively lower rank or low TOC (Kotarba et al., 2002; Varma et al., 2014a, 2015a and b). The calculated vitrinite reflectance (VRo) is between 0.51 to 0.85 % (Table 5). It has been observed that the onset of thermogenic gas generation has occurred in carbonaceous shale samples of middle-upper Pennsylvanian samples which have VRo values of 0.5-0.8%. Organic materials are Type III organic matter (terrigenous vitrinite) indicating that generated hydrocarbons should be gas-prone (Hackley et al., 2009). The fact that VRo values ranging between 0.51 to 0.85% indicate the onset of thermogenic gas generation has been accepted by many (Hunt, 1996; Clayton, 1998). The HI against Calc. VRo% (Figure 16), also confirmed the shales samples are of Type III kerogen, which is prone to generate gas. The HI and calc. VRo% values indicate that the shale beds have good to very good source rock potential for gas generation.

CONCLUSION

Six shale samples from shale beds of Barakar and Barren measure formations in north and south Karanpura coalfields are fine grained, dark to grey colour with low moisture and high inorganic content. Large cumulative thickness of shale beds suggests the potential of economic gas recovery, despite the low *in-situ* gas content of shale beds. *In-situ* gas content compared with adsorption capacity reveals that shale beds in the study area are under to moderately saturated with gas. Methane concentration in desorbed gas is sufficient for easy use of shale gas after recovery from reservoir. Effect of depth and confining pressure on permeability and

porosity indicated that the increasing pressure do not have any impact on shale pores, and fractures and may be due to higher inorganic matter content, which has greater strength. The organic matter is predominantly gas prone Type III kerogen and marginally matured source for gas generation. The various relationships established between geological, geochemical, sorption capacity, TOC and Rock Eval results, porosity and permeability of shale beds in the study area may be useful for shale characterization and better understanding of shale as gas reservoirs. The latest technologies available for shale gas exploration and well completion can be used for economic recovery of shale gas in the study area.

References

- ASTM standards D: 1412-93, (section 5) (1997) Standard test method for equilibrium moisture of coal at 96 to 97 percent relative humidity and 30°C, American Society for Testing and Materials, USA.
- BARKER, C. (1974) Pyrolysis techniques for source rock evaluation. Amer. Soc. Petrol. Geol. Bull., v.58, pp.2349-2361.
- BERTARD, C., BRUYET, B. and GUNTHER, J. (1970) Determination of desorbable gas concentration of coal (Direct method), Internat. Jour. Rock Mechanics and Min. Sci., v.7, pp.43-65.
- BLATT, HARVEY and ROBERT J. TRACY (1996). Petrology: Igneous, Sedimentary and Metamorphic, 2nd ed., Freeman, pp. 281-292. ISBN 0-7167-2438-3.
- BIS, BUREAU OF INDIAN STANDARD NO. 1350 (Part 1), (1984) Methods of Test of Coal and Coke. second Revision, 1995.
- CHEMITO, (2004) Gas chromatograph (GC) operation manual, Chemito Instruments India Pvt.Ltd., 151p.
- CLAYPOOL, G.E. and REED, P.R. (1976) Thermal analysis technique for source rock evaluation: Quantitative estimate of organic richness and effects of lithologic variation. Amer. Soc. Petrol. Geol. Bull., v.60, pp.608-626.
- CLAYTON, J.L. (1998). Geochemistry of coalbed gas—a review. Internat. Jour. Coal Geol., v.35, pp.159-173.
- CLEMENTZ, D.M., DEMAISON, G.J. and DALY, A.R. (1979) Well site geochemistry by programmed pyrolysis, Proceedings of the 11th Annual Offshore Technology Conference, Houston, OTC 3410, v.1, pp.465-470.
- CMPDIL, (1993) Coal Atlas of India, 1st ed., Central Mine Planning and Design Institute Ltd., pp.92-95.
- CROSDALE, P.J. (2000) Instruction Manual: CGRI Adsorption Isotherm Determination Apparatus by Volumetric Method, pp.1-58.
- CORNFORD, C., GARDNER, P. and BURGESS, C. (1998) Geochemical truths in large data sets: I. Geochemical screening data: Organic Geochemistry, v.29(1-3), pp.519- 530.
- DANIEL, J.K. Ross and BUSTIN, R. MARC (2009) The importance of shale composition and pore structure upon gas storage potential of shale gas reservoirs. Original Research Article Marine and Petroleum Geology, Volume 26, Issue 6, June 2009, pp. 916-927.

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- DIAMOND, W.P. and LEVINE, J.R. (1981) Direct method for determination of the gas content of coal: procedure and results, US Bureau of Mines Report of Investigation, RI 8515.
- EKWEOZOR, C.M. and GORMLY, J.R. (1983) Petroleum geochemistry of LateCretaceous and Early Tertiary Shales penetrated by Akukwa-2 well in the AnambraBasin, Southern Nigeria. Jour. Petrol. Geol., v.6, pp.207-216.
- EIA, U.S. ENERGY INFORMATION ADMINISTRATION (2015) Annual Energy outlook 2015, April 2015, http://www.eia.gov/ forecasts/aeo/mt naturalgas.cfm
- EIA, U.S. ENERGY INFORMATION ADMINISTRATION (2011), World Shale Gas Resources: An Initial Assessment of 14 Regions Outside the United States, Washington.
- ESPITALIE, J., LAPORTE, J.L., MADEC, M., MARQUIS, F., LEPLAT, P. and PAULET, J., (1977). Methoderapide de caracterisation des roches meres, de leur potential petrolieret de leur degree d'evolution. Rev. Inst. Petr., v.32, pp.23-45.
- ESPITALIE, J., DEROO, G. and MARQUIS, F. (1985). La pyrolyse rockevaletses applications. Rev. Inst. Fr. Pet., v.40, pp.563–579 and pp.755–784.
- ESPITALIE, J. MARQUIS, F., and BARSONY, I. (1984) Geochemical logging *In:* K.J. Voorhees (Ed.), Analytical pyrolysis – Techniques and applications. Boston, Butterworth, pp.276-304.
- FENG, K.K., CHENG, K.C. and AUGSTEN, R., (1984). Preliminary evaluation of the methane production potential of coal seams at Green-hills mine, Elkford, British Columbia, CIM Bull., no.77, pp.56-60.
- GSI, (1977) Coal Resources of India. Mem. Geol. Surv. India, v.88, pp.155-177.
- HACKLEY, P.C., GUEVARA, E.H., HENTZ, T.F. and HOOK, R.W. (2009) Thermal maturity and organic composition of Pennsylvanian coals and carbonaceous shales, north-central Texas: implications for coalbed gas potential. Internat. Jour. Coal Geol., v.77, pp.294-309.
- HAKIMI, M.H., SHALABY, M.R. and ABDULLAH, W.H. (2012a). Molecular composition and organic petrographic characterization of Madbi source rocks from the Kharir Oilfield of the Masila Basin (Yemen): palaeoenvironmental and

maturity interpretation. Arab. Jour. Geosci., v.5, pp.817-831.

- HAKIMI, M.H., SHALABY, M.R. and ABDULLAH, W.H. (2012b) Geochemical and petrographic characterization of organic matter in the Upper Jurassic Madbi shale succession (Masila Basin, Yemen): origin, type and preservation. Org. Geochem. v.49, pp.18–29.
- HAKIMI, M.H., ABDULLAH, W.H., SIA, S.G. and MAKEEN, Y.M. (2013) Organic geochemical and petrographic characteristics of Tertiary coals in the northwest Sarawak, Malaysia: implications for palaeoenvironmental conditions and hydrocarbon generation potential. Mar. Pet. Geol., v.48, pp.31-46.
- HORSFIELD, B., (1985). Pyrolysis studies in petroleum exploration, *In:* J. Brooks and D. Welte (Eds.), Advances in Petroleum Geochemistry, v.1, pp.247-298.
- HUNT, J.M. (1996) Petroleum Geochemistry and Geology. 2nd ed. W.H. Freeman and Company, New York, 743p.
- JARVIE, DANIEL M., HILL, RONALD J. and POLLASTRO, RICHARD M. (2005). Assessment of the Gas Potential and Yields from Shales: the Barnett Shale Model. *In:* Cardott, B.J. (Ed.), Unconventional energy resources in the southern Midcontinent, 2004 symposium: Oklahoma Geological Survey Circular 110, pp.37-50.
- JARVIE, DANIEL M., RONALD J. HILL, TIM E. RUBLE and RICHARD M. POLLASTRO, (2007) Unconventional shale-gas systems: The Mississippian Barnett Shale of north-central Texas as one model for thermogenic shale-gas assessment. AAPG Bull., v.91(4) (April 2007), pp.475-499.
- JUDE E. OGALA, (2011) Hydrocarbon potential of the upper cretaceous coal and shale units in the Anambra basin, Southeastern Nigeria. Petroleum & Coal, v.53(1), pp.35-44.
- KAWATA, Y. and FUJITA, K. (2001). Some Predictions of Possible Unconventional Hydrocarbons Availability until 2100. Paper SPE 68755, presented at the SPE Asia Pacific Oil and Gas Conference, Jakarta, 17–19 April. DOI: 10.2118/68755-MS.
- KISSEL, F.N., MCCULLOCH, C.M. and ELDER, C.H. (1973) The direct method of determining methane content of coalbeds for ventilation design, US Bureau of Investigation, RI 8043.
- KOTARBA, M., CLAYTON, J., RICE, D. and WAGNER, M., (2002). Assessment of hydrocarbon source rock potential of Polish bituminous coals and carbonaceous shales. Chem. Geol., v.184, pp.11–35.
- LANGFORD, F.F. and BLANC-VALLERON, M.-M. 1990) Interpreting Rock-Eval pyrolysisdata using graphs of pyrolizable hydrocarbons versus total organic carbon. AAPG Bull., v.74, pp.799-804.
- LARTER, S.R. and DOUGLAS, A.G. (1982) yrolysis methods in organic geochemistry: An overview. Jour. Anal. Appld. Pyrol., v.4, pp.1-19.
- McCulloch, C.M., LEVINE, J.R., KISSEL, F.N. and DEUL, M. (1975) Measuring the methane content of bituminous coalbeds, US Bureau of Mines Report of Investigation, RI 8043.
- MENDHE, V.A., MISHRA, P. and VARADE, A.M. (2010) Coal Seam Reservoir Characteristics for Coalbed Methane in North and South Karanpura Coalfields, Jharkhand in Sedimentary Basins of India: Recent Developments; Gondwana Geol. Mag., Spec. Vol., No.12, pp.141-152.

- MENDHE, V.A. (2014) Coalbed methane and shale gas potential in India, Sustainable Environment and Role of Clean Coal Energy: Edited book by S.K.Mangal and Sanjay Gupta; published by Center for Research in Rural and Industrial Development (CRRID), Chandigarh, pp.165-185.
- MoPNG, (2013) Policy for the exploration and exploitation of shale gas inIndia'. The Ministry of Petroleum & Natural Gas, India. September, 2013.
- NICHOLAS, B.H., KATHERINE, H.F., RICHARD, D.P., TIMOTHY, S.W. and GARETH, D.M. (2004) The character and origin of lacustrine source rocks in the Lower Cretaceous synrift section, Congo Basin, West Africa. AAPG Bull., v.88, pp.1163-1184.
- PETERS, K.E. and CASSA, M.R. (1994) Applied source rock geochemistry. *In:* Magoon, L.B., Dow, W.G. (Eds.), The Petroleum System from Source to Trap. AAPG Mem. v.60, pp.93-120.
- PETERS, K.E. (1986) Guidelines for evaluating petroleum source rock using programmed pyrolysis, AAPG Bull., v.70(3), pp.318-329.
- PETERS, K.E. and SIMONET, B.R.T. (1982) Rock-Eval pyrolysis of Quaternary sediments from Leg 64, sites 479 and 480, Gulf of California. Initial Report of the deep Sea Drilling Project, v.64, pp.925-931.
- RAO, V.K. (2013) Shale Gas Exploration in India Potential, Challenges and Opportunities, Infraline Energy- Business Report Series, February 28, 2013.
- RICE, D.D., CLAYTON, J.L. and PAWLEWICZ, M.J., (1989). Characterization of coal-derived hydrocarbons and source-rock potential of coal beds, San Juan basin, New Mexico and Colorado, USA. Internat. Jour. Coal Geol., v.13, pp.597-626.
- TEERMAN, S. and HWANG, R. (1991) Evaluation of the liquid hydrocarbon potential of coal by artificial maturation techniques. Org. Geochem., v.17, pp.749–764.
- TEMCO (2003) Operation user manual for porosity and permeability determination. Temco Controls Ltd, USA.
- VARMA, A.K., HAZRA, B., CHINARA, I., MENDHE, V.A., DAYAL, A.M. (2015a) Assessment of organic richness and hydrocarbon generation potential of Raniganj basin shales, West Bengal, India. Mar. Pet. Geol., v.59, pp.480–490.
- VARMA, ATUL KUMAR, SRICHANDAN BISWAL, BODHISATWA HAZRA, VINOD ATMARAM MENDHE, SATABDI MISRA, SURESH KUMAR SAMAD, BHAGWAN D. SINGH, ANURODH MOHAN DAYAL and DEVLEENA MANI (2015b) Petrographic characteristics and methane sorption dynamics of coal and shaly-coal samples from Ib Valley Basin, Odisha, India. Internat. Jour. Coal Geol., v.141–142 (2015), pp.51–62.
- VARMA, A.K., HAZRA, B., SAMAD, S.K., PANDA, S. and MENDHE, V.A. (2014a) Methane sorption dynamics and hydrocarbon generation of shale samples from West Bokaro and Raniganj basins, India. Jour. Nat. Gas Sci. Engg., v.21, pp.1138–1147.
- VARMA, A.K., HAZRA, B., SAMAD, S.K., PANDA, S., MENDHE, V.A. and SINGH, S. (2014b) Shale gas potential of Lower Permian Shales from Raniganj and West Bokaro Basins. India. 66th Annual Meeting and Symposium of The International Committee for Coal and Organic Petrology (ICCP-2014), pp.40–41.

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