Petrophysical Characterization of Gondwana Shales of South Karanpura Coal Field, Jharkhand, India.

Piyush Sarkar*
Department of Earth Sciences
IIT Bombay
Mumbai, India
sarkar.piyush@iitb.ac.in

Kumar Hemant Singh
Department of Earth Sciences
IIT Bombay
Mumbai, India
kumar.h.singh@iitb.ac.in

SUMMARY

Sedimentary rocks such as shales are the most abundant sedimentary rocks in the earth's crust. Shales are characterized by preferred particle orientations of platy clay minerals, strong laminations and presence of fissility. In petroleum geology, organic shales behave as both source rocks as well as seal rocks that trap oil and gas. In seismic exploration, shales interface with other rocks to form good reflectors. As a result, seismic and petrophysical properties of shales and the relationships among these properties are important for both exploration and reservoir management. The Gondwana shale of South Karanpura coalfield, Eastern India of Barakar (lower Permian) and Barren-Measures (Middle Permian) formation, majorly characterized by non-marine sedimentary fill and narrow graben structures, are used for petrophysical characterization. Because of thermal maturity and high content of organic matter in shale rock, South Karanpura coalfield is considered as one of the potential shale gas field in Damodar Valley Basin and it is a part of the "Gondwana" basins of Eastern India. This paper discusses various experimental techniques that are applied to shales to obtain velocity, elastic properties structure, minerals composition, texture/fabric and pore types. These techniques include ultrasonic acoustic measurements, X-ray diffraction (XRD), and scanning electron microscopy (SEM). The frequency values used for acoustic measurements are 54 KHz (P-wave) and 250 KHz (S-wave). The resolution of these imaging techniques varies from micrometres to angstroms and the resulting images generally reflect the composition, topography, or combination of both. The estimated P-wave velocity for Barren-Measures and Barakar formation varies from 1463-1890m/s and 2110-3947m/s, respectively while the Shear-wave velocity varies from 898m/s-1232m/s to 1306m/s-2515m/s respectively which suggest that formation is hard and compact in the latter case. XRD and SEM analysis reveals the presence of clay minerals and other minerals, organic matter, texture/topography and pore types in the shale rock sample. The clay minerals identified consist of Kaolinite, Illite while Quartz, Siderite, Muscovite, Orthoclase and Rutile comprises of non-clay minerals in the sample.

Key words: P and S wave, XRD, SEM, Elastic Moduli, Minerals, Pores types.

INTRODUCTION

The presence of organic matter type and degree of thermal maturity in shale makes them ideal source rock for hydrocarbon generation. The shale rock containing more than 1% Total Organic Carbon (TOC) has effective source potential to produce adequate amount of oil and gas (Hunt, 1995). Large amounts of hydrocarbon have been produced from marine shales from pre-Cambrian to Tertiary in age (Dyni, 2006) such as the Bakken shale, Eagle Ford shale (Jarvie, 2012) and Mowry Shale (Finn, 2007). These shales are characterized by their high TOC and thermal maturity and type of organic matter content. Similar to the shales deposited in marine environment, non-marine shales of high TOC and thermal maturity are also potential source of hydrocarbon. The Barren Measures shale of Permian age is dominated by type III kerogen and it is predicted as the shale gas prospective horizon based on higher content of total organic carbon (3.05-9.38%) (Tewari and Dutta, 2014), thickness (upto 1000m), and higher degree of thermal maturity (>1%). Barakar Formation also has high organic content and thermal maturity value than Barren Measures formation (Mukhopadhyay *et al*, 2010).

The ultrasonic compressional-wave and shear-wave velocities (McSkimin, 1964), powdered X-ray diffraction (XRD) and scan electron microscopy (SEM) of shale rock measured in laboratory give a useful basis for understanding the geological significance of observed variations in seismic-wave velocities, mineralogy and surface texture/topography. The physical properties of rock measured in the laboratory are necessary input parameters for models that help analyze and predict the response of rock masses in many areas of engineering geology. The laboratory samples are usually very small and homogeneous compared to the subsurface rock mass which contains discontinuities and inhomogeneities. Nevertheless, the laboratory tests on samples that are small and intact can still provide useful information, if the discontinuities can be characterized independently. So, a composite picture of rock mass behavior can be assembled based on intact material plus discontinuity properties. Ultrasonic velocities have been used to estimate elastic moduli. These elastic moduli of rock are frequently used in the study of geotechnical problems. X-ray powder diffraction is a fast and reliable technique used for the routine identification and quantification of all minerals (Moecher, 2004) and mixture or intergrowth of minerals present in clay-rich rocks (shale) which may not be revealed to analysis by any other technique. Accurate quantitative mineral analysis is important in petrophysics, rockphysics, petrological studies, and in industrial applications of rocks that contain clay minerals such as coal and petroleum assessments, mineral-resource assessments, remote sensing to mention a few. XRD analysis also gives quantitative volume fractions of minerals in a sample. Scan electron microscopy on the contrary is used to analyze the surface topography or surface texture as well as characterization of pore types of shale rock.

The Study Area

Figure 1 shows the location map of the South Karanpura coalfield, in Ramgarh district, Jharkhand state of India. The South Karanpura coalfield extends from 23.65^0 to 23.73^0N and from 85.29^0 to 85.48^0E. The shale rocks found here belongs to Gondwana supergroup. The samples of shale collected belong to two formations. Five samples are of Barakar formation which are lower Permian in age while another five samples are mid-Permian from Barren Measures formation (De *et al*, 2003). The core samples of shale rock were taken from two different bore holes from different depths of Urimari block of South-Karanpura coal field. There are lots of surface and underground coal mines on the South Karanpura coalfield which include mines in the Barka Sayal Area (Saunda, Central Saunda, Bhurkunda, Sayal, Urimari, and North Urimari) and in the Argada Area (Gidi and Sirka). Urimari block is one of the several underground coal mine. The network of coal mines in the South Karanpura coalfield is owned by Central Coalfields Limited (a subsidiary of Coal India).

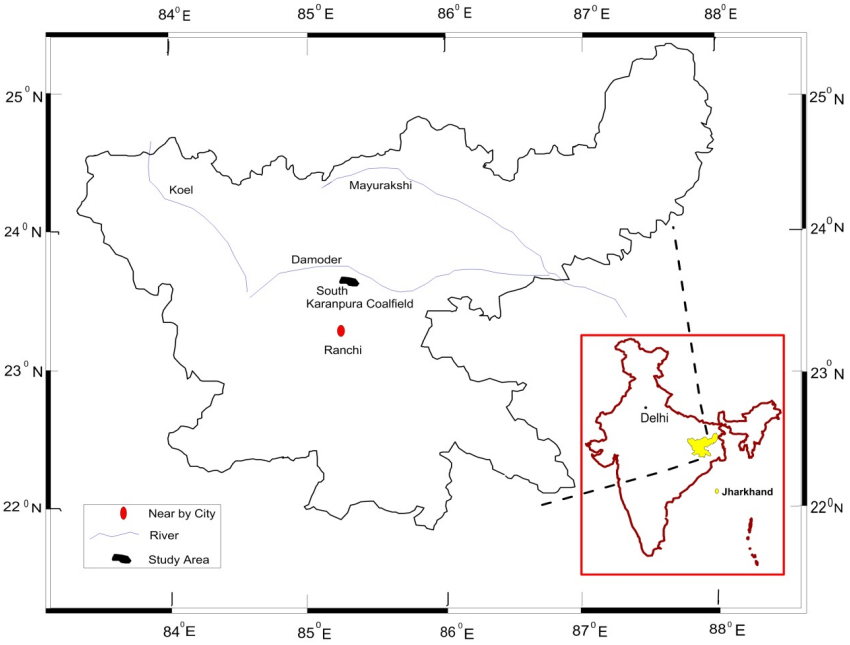


Figure 1: Map showing the location of study area (black region).

LABORATORY MEASUREMENTS

Three methods were used to characterize the shale rock samples of South Karanpura coalfield. These include ultrasonic acoustic measurements, X-Ray Diffraction (XRD) and Scan Electron Microscopy (SEM). Five core samples each from Barakar and Barren-Measures formations taken from two boreholes of Urimari block of South Karanpura coalfield are assigned with the following naming convention in order to identify the specimen depth and formation.

For Barren-Measures formation (Bn):
Shale Samples (SK): 2_SK_Bn_21m, 2_SK_Bn_23m, 2_SK_Bn_47m, 2_SK_Bn_53m, 2_SK_Bn_59m.

For Barakar Formation (Br):
Shale Samples (SK): 1_SK_Br_53m, 1_SK_Br-95m, 1_SK_Br-137m, 1_SK_Br-156m, 1_SK_Br-325m.

Here in the naming convention, the last two numbers represents the depth from which the core samples were retrieved.

Ultrasonic Velocity Measurement

The data on elastic properties of shales are insufficient for two reasons. First, obtaining and preserving shale samples is difficult and second, taking measurement is a time consuming process (Vernik et al., 1997). Before the ultrasonic measurement, the core samples are smoothened to level the top and bottom part of the sample so that the transducer could make good contact with the surface. The length of the core sample and the diameter of its cross-section is measured with the help of highly sensitive digital Vernier Caliper. To obtain elastic constants, ultrasonic velocity measurements are performed on dry core samples of shale using transducers of frequency 54 KHz for P (compressional) wave and 250 KHz for S (Shear) wave at room temperature and pressure. The weight and dimensions of the samples are measured which is used to compute the density of the core sample by taking the ratio of weight and volume of the sample.

The laboratory velocities are measured using ultrasonic equipment (Pundit plus) by measuring in the direction perpendicular to the bedding plane of rock samples (Figure 2). Using the velocities and density value, the elastic moduli are calculated with the standard equations as presented in ASTM (1995).

$$E = \frac{\rho V_s^2 (3V_p^2 - 4V_s^2)}{(V_p^2 - V_s^2)} \quad (1)$$

$$K = \frac{\rho (3V_p^2 - 4V_s^2)}{3} \quad (2)$$

$$\mu = \rho V_s^2 \quad (3)$$

Where E is Young's Modulus, μ is Modulus of rigidity or shear modulus and K is Bulk modulus. Vp and Vs are compression and shear waves velocities and ρ is density. The velocities and the elastic moduli thus measured are plotted against depth (Figure 4 and 5). The Figure 3 shows one of the snap shots taken during ultrasonic measurement while using the equipment (Pundit plus).

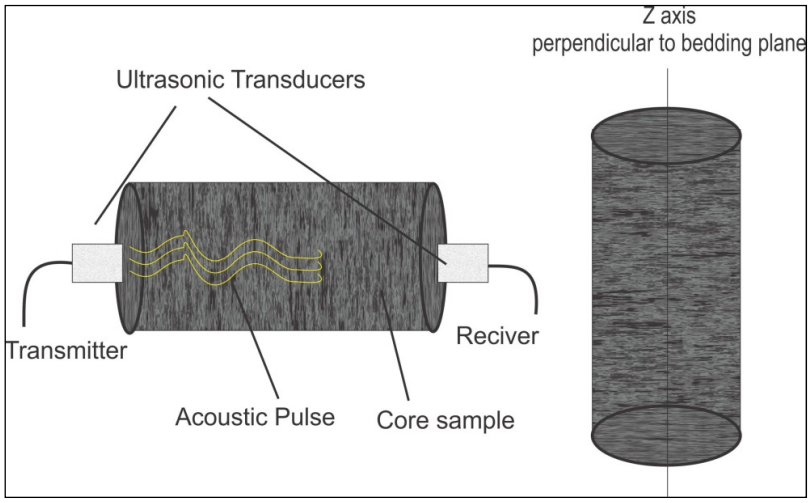


Figure 2: Schematic diagram of Ultrasonic measurement on core sample.

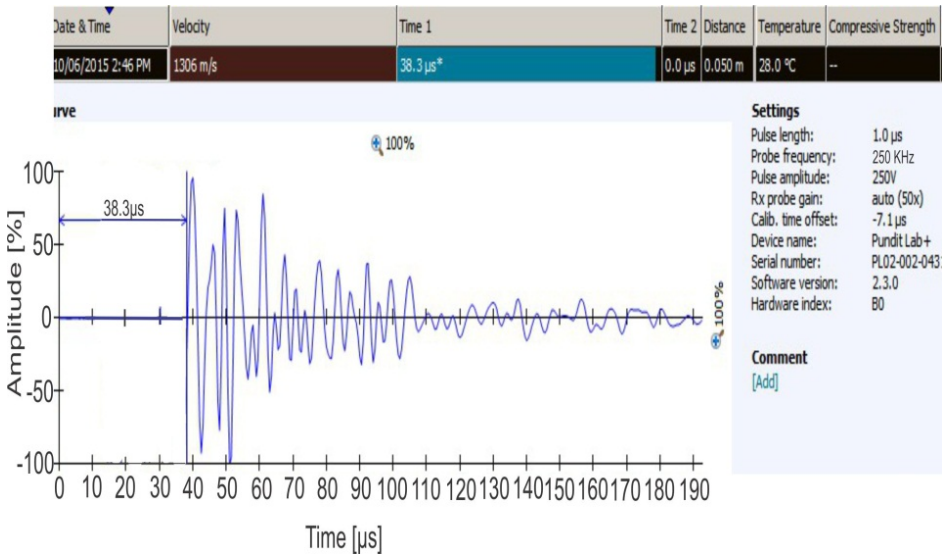


Figure 3: Snap shot showing the ultrasonic measurement of S wave of core sample (1_SK_Br_53m) plotted as Amplitude (percentage) versus Time (microsecond).

Powder X-ray Diffraction (XRD)

Using the X-ray diffractometer (PANalytical 'Empyrean') in the XRD laboratory, the powdered samples of shale is investigated to identify the different minerals including clay minerals present in shale (Moore *et al,* 1997*,* Srodon *et al*, 2001). Powder XRD technique is used for identification of different crystalline materials in the rock formation. Each mineral has its own unique fingerprint.

The following steps are involved during the sample preparation for X-Ray diffraction test:

a) Obtain a few tenth of a gram (or more) of the material.
b) Grind the sample to a fine powder with agate mortar and pestle. Powder with less than ~10 μm (or 200-mesh) in size is preferred.
c) Wash the 200 mesh brass filter with double distilled water and dry it in oven for 15 minutes before filtering the powder and repeating the above process for every sample.

The sample and detector rotate about their respective angles which help record the intensity of diffracted X-rays. A peak in intensity occurs when the mineral contains lattice planes with d-spacings appropriate to diffract X-rays at that value of θ. The 2θ position of the diffraction peak is typically measured as the center of the peak. The d-spacing of each peak is then calculated with the help of Bragg equation for the appropriate value of λ (Bragg, 1913). After that an automated search/match routines compare the d-spacing of the unknown to those of known materials from the database. Since each mineral has a unique set of d-spacings, matching these d-spacings helps to identify the minerals in the sample. The data is processed using HIGH SCORE PLUS 4.0 software.

Scan Electron Microscopy (SEM)

The Scanning electron microscopy (JSM '6930' JEOL) is used to generate a two-dimensional raster image by bombarding the surface of a sample with electrons beam and detecting the scattered electron beams (Goldstein, 2012; Reimer, 2000). SEM helps in analyzing topographic characteristics and atomic composition. This technique is capable of producing very high-resolution images showing micrometer-scale to nanometer features, grain size, pore size, pore types, minerals grains and bulk mineral composition that are present in the sample.

The Shale Rock samples prepared for SEM analysis are obtained by gently and carefully breaking a small piece or chip of rock from the core sample with a small rock-chopper or knife. The surface of rock should be fresh surface and for minimizing the contamination, it is cleaned with a solvent such as acetone. Next, the samples are dried in low temperature oven for 5-6 hours. The size of rock sample is typically of dimension 5x5x2 mm. The sample is then coated on 'Lieca ACE 200' with a conductive metal, such as gold, by sputter coating technique. For getting best results, samples are carefully handled with gloves and tongs because skin oil from fingers often contaminates the sample which produces out-gas in the SEM vacuum system thereby degrading the SEM image. The sample is attached to a SEM specimen plug with adhesive and a thin line of silver paint is coated on the sample in order to provide an electrical ground to the sample to the plug. This coating of 200A° is required in order to get a clear image of non-conducting rock sample. After coating, the sample is ready for SEM analysis.

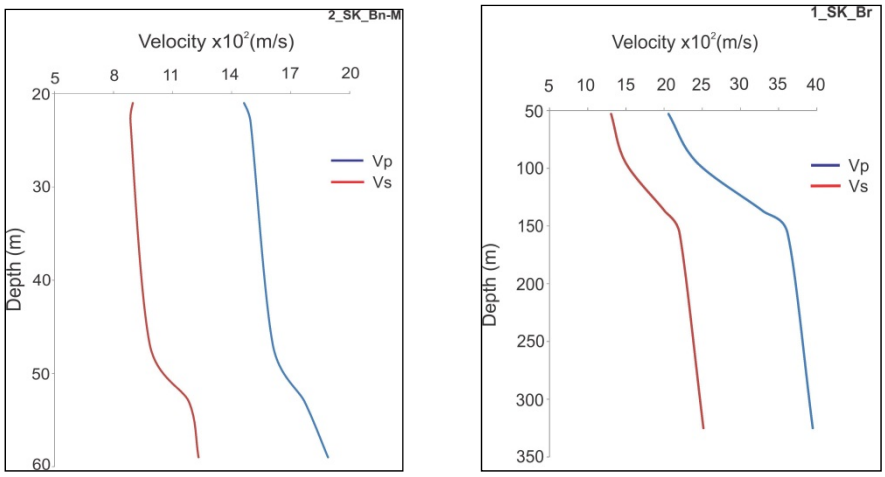
RESULTS

Ultrasonic Velocity

The compressional and shear wave velocities thus measured is presented in Table 1. The measured velocities plotted against the depth for Barren-Measures and Barakar formations of South Karanpura coalfield are shown in Figures 4(a) and 4(b), respectively. The elastic moduli are calculated using ultrasonic velocities as input for equation 1, 2 and 3 described above and plotted against depth for Barren-Measures and Barakar formation of South Karanpura coalfield shown in Figures 5(a) and 5(b), respectively.

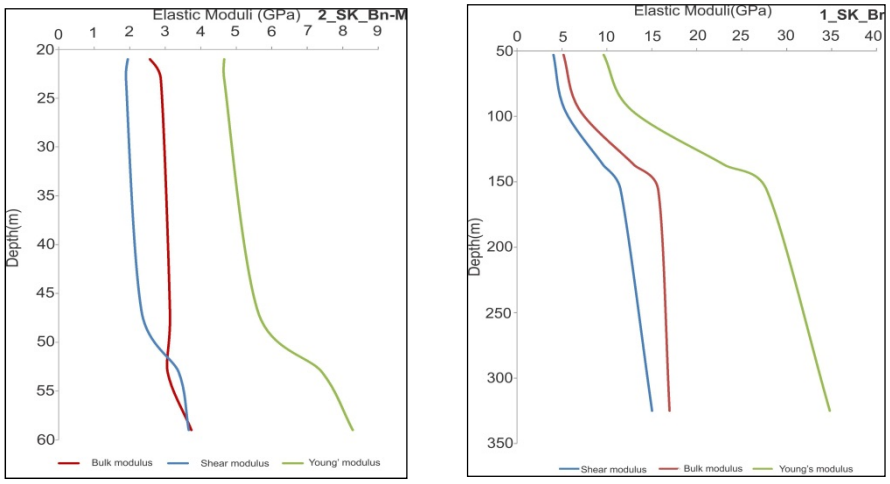
2_SK_Bn-M			1_SK_Br		
Depth(m)	**Vp (m/s)**	**Vs(m/s)**	**Depth(m)**	**Vp (m/s)**	**Vs(m/s)**
21	1463	898	53	2110	1306
23	1496	886	95	2430	1495
47	1610	985	137	3293	2007
53	1770	1182	156	3616	2202
59	1890	1232	325	3947	2515

Table 1: Ultrasonic P and S wave velocities measured from core samples of shale recovered from different depths of Barren-Measures and Barakar formation.



(a) **(b)**

Figure 4: Variation of P and S wave with depth for (a) 2_SK_Bn-M and (b) 1_SK_Br.



(a) **(b)**

Figure 5: Variation of Young's, Bulk and Shear modulus with depth for the formation (a) 2_SK_Bn-M and (b) 1_SK_Br.

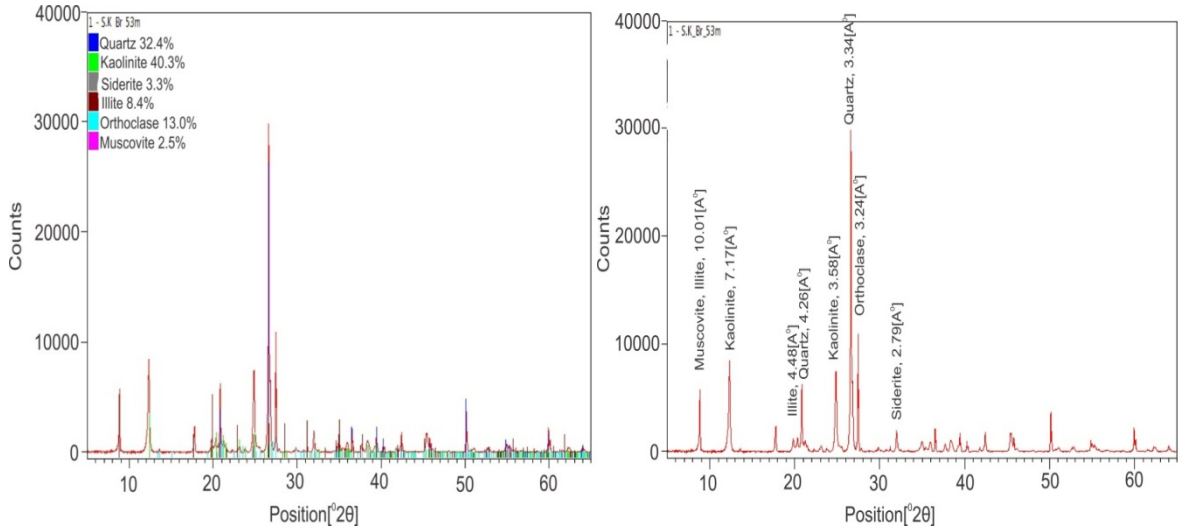
X-Ray Diffraction (XRD)

The results from X-ray diffraction are presented as X-ray counts (i.e. intensity) v/s peak positions (Figure 6(a)). The counts or intensity (I) is documented as peak height intensity or intensity above background. The following results are obtained by XRD. Clay minerals and other minerals are identified in the shale rock sample. The identified clay minerals consist of Kaolinite, Illite and non-clay mineral such as Quartz, Siderite, Muscovite, Orthoclase and Rutile in the sample. All of these minerals are identified due to their unique fingerprints (Figure 6(b)). The primary 2θ values for minerals (Breeden, 2004) present in shale are listed in Table 2.

The samples from both the formations contain clay minerals (Kaolinite + Illite) approximately 40.0% to 55.0% whereas quartz is approximately 30.0% to 40.0%. The other minerals such as Muscovite, Orthoclase and Siderite are approximately 10.0% - 20.0%, 1.4% - 13.0% and 0.2% - 3.3% in the given samples, respectively (Tables 3 and 4). Another mineral named Rutile (~ 1.0%) has been identified in the sample at a depth of 59m.

Minerals	2θ [degree]
Quatrz	20.9, 26.6
Kaolinite	12.3, 24.8
Illite	8.8, 19.8
Siderite	31.9
Muscovite	8.8
Orthoclase	27.4
Rutile	36.05

Table 2: Shale minerals and their 2θ values (Breeden, 2004).



(a) **(b)**

Figure 6: (a) XRD diffraction patter and (b) mineral signature with d-spacing value for shale rock sample 1_SK_Br_53m.

Minerals / Depth(m)	Wt.%				
	Quartz	Clay (Kaolinite+Illite)	Siderite	Muscovite	Rutile
21	34.9	53.7	1.6	9.8	-
59	36.6	41.2	0.7	20.6	0.9

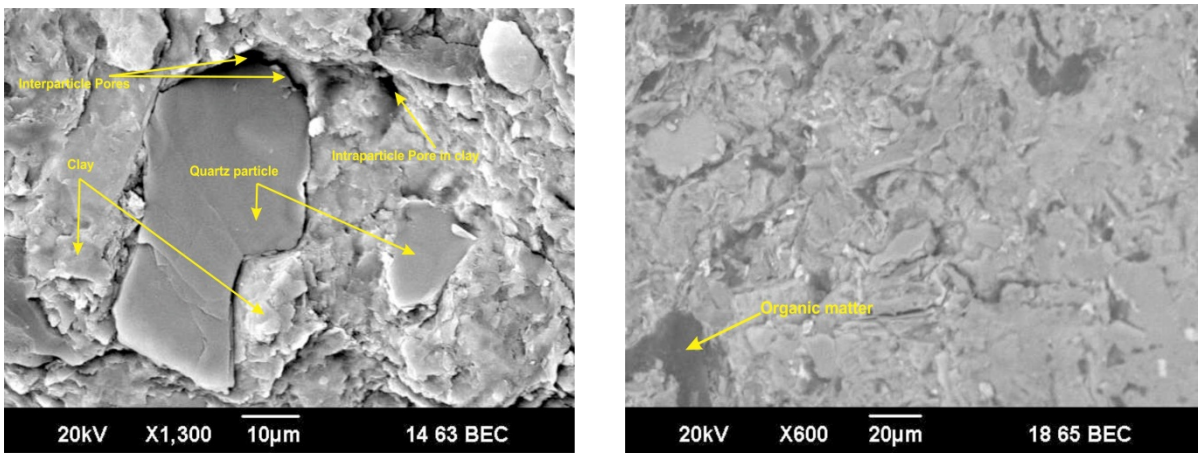
Table 3: Weight % of different minerals in the samples for the formation 2_SK_Bn-M at different depths.

Minerals / Depth(m)	Wt.%				
	Quartz	Clay (Kaolinite+Illite)	Siderite	Muscovite	Orthoclase
53	32.4	48.7	3.3	2.5	13.0
137	32.4	54.9	0.2	11.3	1.4
325	40.8	38.3	2.0	18.9	-

Table 4: Weight % of different minerals in the samples for the formation 1_SK_Br at different depths.

Scan Electron Microscopy (SEM)

SEM images shows texture and topography of the shale rock surface (Figure 7). Figure 7(a) shows quartz grain embedded in flaky clay particle. In oil and gas shales, pore size and connectivity are important for the storage and flow of hydrocarbons. Two types of pores: intraparticle pore and interparticle pores are analysed in this study (Loucks *et al*., 2012). Interparticle pores are found between clay flakes or at the edges of quartz, or other crystals while intraparticle pores are found within clay grains (Figure7 (a)). Figure 7(b) shows presence of organic matter in shale sample. The size of pores in the given samples varies from nanometre to few micrometre.



(a) **(b)**

Figure 7: SEM images displaying (a) shale minerals and pore types. Interparticle pores along boundary of quartz and clay grains and Intraparticle pores within clay grains. (b) Presence of organic matter in the shale.

CONCLUSIONS

This paper discusses the measured parameters from three laboratory methods used in the study: Ultrasonic velocity, XRD and SEM. The results are used to characterize the Gondwana shale rock of South Karanpura coalfield. Ultrasonic measurements show the acoustic velocity variation in shale with depth. Both P-wave and S wave velocity increases with depth. It is inferred that the increase in velocity is due to increase in compactness of the formation. The estimated elastic parameter also increases with depth. X-ray diffraction results show the presence of mineral types in the sample. Minerals Quartz, Kaolinite, Illite, Muscovite, Siderite, Orthoclase, and trace of Rutile are identified in samples from both formations. Quartz shows larger peaks indicating that it has good crystalline form. On the other hand, clays does not diffract as good as Quartz because they lack good and uniform crystalline form and therefore, their peaks are lower than of quartz. There are no swelling clays (Bentonite, Smectite,) reported in the sample. Scan electron microscopy reveals the presence of platy clay minerals aligned along the bedding plane and Quartz mineral and also the pore types i.e. interparticle pore and intraparticle pore. The velocity of Barakar formation is higher than the Barren Measures formation which suggests that the shale sample from Barakar formation is more compact and hard. In addition the mineral Rutile although found in trace quantity is only found in the samples obtained from Barren-Measures formation.

ACKNOWLEDGMENTS

The author would like to thank various laboratories at the Department of Earth Sciences, Indian Institute of Technology Bombay, Mumbai for providing adequate facilities to carry out these highly sensitive scientific experiments. The author also expresses his gratitude towards Prof. T. N. Singh for allowing us to use the Rock Science and Rock Engineering lab for conducting ultrasonic experiment while Prof. S. Dutta for kindly providing the shale samples.

REFERENCES

ASTM. 1995. Standard test method for laboratory determination of pulse velocities and ultrasonics elastic constants of rocks, D. 2845-95. Annual Book of A.S.T.M. Standards, 4.08. American Society for Testing and Materials, West Conshocken, PA.

Bragg, W.L., 1913, The structure of some crystals as indicated by their diffraction of X rays. Proc. Roy. Soc. Lond, Ser. A 89 (610): 248-277. Bibcode 1913 RSPSA. 89..248B. DOI : 10.1098/rspa.1913.0083.

Breeden, D., and Shipman, J., 2004, Shale analysis for mud engineers: AADE, pages 1–17.

De, A., De, B. and Bera, S., 2003. First Record of Raniganjia Bengalensis (Rigby) Pant & Nautiyal from Lower Permian Beds (Barakar Formation) of South Karanpura Coalfield, Jharkhand, India. Geological Society of India, 61(4), pp.487-490.

Dyni, J.R., 2006. Geology and resources of some world oil-shale deposits(pp. 2005-5294). US Department of The Interior, US Geological Survey.

Finn, T.M., 2007. Source rock potential of upper cretaceous marine shales in the Wind River Basin, Wyoming: Chapter 8 in Petroleum systems and geologic assessment of oil and gas resources in the Wind River Basin Province, Wyoming (No. 69-J-8). US Geological Survey.

Goldstein, J., Newbury, D.E., Echlin, P., Joy, D.C., Romig Jr, A.D., Lyman, C.E., Fiori, C. and Lifshin, E., 2012. Scanning electron microscopy and X-ray microanalysis: a text for biologists, materials scientists, and geologists. Springer Science & Business Media.

Hunt John M., 1995, Petroleum Geochemistry and Geology, Second Edition, W. H. Freeman and Company, New York.

Jarvie, D. M., 2012. Shale resource systems for oil and gas: part 2- shale-oil resource systems. In: Breyer, J.A. (Ed.), Shale reservoirs-giant resources for the 21st century. AAPG Memoir 97, 89-119.

Loucks, R.G., Reed, R.M., Ruppel, S.C. and Hammes, U., 2012. Spectrum of pore types and networks in mudrocks and a descriptive classification for matrix-related mudrock pores. AAPG bulletin, 96(6), pp.1071-1098.

McSkimin, H.J., 1964. Ultrasonic methods for measuring the mechanical properties of liquids and solids. Physical Acoustics, 1(part A), pp.271-334.

Moecher, D.P., 2004. Characterization and identification of mineral unknowns: A mineralogy term project. Journal of Geoscience Education,52(1), p.5.

Moore, D.M., and Reynolds, R.C., Jr., 1997. X-ray Diffraction and the Identification and Analysis of Clay Minerals (2nd ed.): Oxford (Oxford Univ. Press).

Mukhopadhyay, G., Mukhopadhyay, S.K., Roychowdhury, M. and Parui, P.K., 2010. Stratigraphic correlation between different Gondwana basins of India. Journal of the Geological Society of India, 76(3), pp.251-266.

Reimer, L., 2000. Scanning electron microscopy: physics of image formation and microanalysis, IOP.

Środoń, J., Drits, V.A., McCarty, D.K., Hsieh, J.C. and Eberl, D.D., 2001. Quantitative X-ray diffraction analysis of clay-bearing rocks from random preparations. Clays and Clay Minerals, 49(6), pp.514-528.

Tewari, A. and Dutta, S., 2014. Organic Geochemistry And Pore System Characterization Of Gas Shales: An Example From The Lower Gondwana Shales, Eastern India.

Vernik, L., and Liu, X., 1997, Velocity Anisotropy in Shales: A Petrophysical Study, Geophysics, 62, 521-532.