

Chapter 3:Geology of Barren Measures Formation

Introduction

Earlier research works endorsed the geological controls on shale gas generation, accumulation and production. The depositional environments and tectonic events on sedimentation can cause great impact to mineralogy, geochemical and geotechnical characteristics of shale rock properties. Therefore, this chapter is written to discuss the lithological characters, mineral composition, texture etc. of Barren Measures shales of Chalbalpur-Mahishmura area of Raniganj Field. An intuition into regional geology, lithostratigraphy, tectonic setting etc. helps in understanding the Permian sediments of the Raniganj Field. Within the study area, the exposures are very scanty and very much restricted to nala section. The author could observe and measure few sections only. However, the geology of this area was mainly brought out from the subsurface data collected from the boreholes of Coal India Ltd, which covers the entire Barren Measures. It is therefore, in the present investigation, available borehole lithologs (13 boreholes i.e. R#1, R#2, R#3, R#4, R#5, R#6, R#7, R#8, R#9, B#1, B#2, B#3 and B#4) were analyzed and correlated to understand the lateral continuity of Barren Measures Formation as well as the subsurface geology of the study area. The core samples were collected from four boreholes (B#1, B#2, B#3 and B#4) and systematically analyzed using Polarizing Microscope, XRD, SEM, EDX techniques and an attempt has been made to bring out the geology of the Barren Measures Formation.

Geological Settings and Lithostratigraphy

The Gondwana basins in India are intra- cratonic in nature and surrounded by Pre-Cambrian terrains. Raniganj field is in the easternmost part of Damodar Basins (Chakraborty *et al*, 2003). The Field has typical half-grabben E-W trending and westerly plunging synform. Depositional environment of the basin was predominantly terrestrial (fluvial and lacustrine) with abundant Glossopteris, Gangampteris flora (Veevers and Tewari, 1995).

The Gondwana sediments consist of Talchir, Barakar, Barren Measures and Raniganj Formations overlain on the metamorphic basement (Geological map of Raniganj Field Figure 3.1). The southern part of the Raniganj Field is bounded by the southern boundary fault (SBF). The southern boundary faults controlled the growth of half graben type of the basin with an increasing thickness of the sediments towards the south (Ghosh, 2002). The eastern margin of the Raniganj Field is bounded by a zone of NNW–SSE trending normal faults. Several intrabasinal faults (Figure 3.1) are present and are dipping towards NE and NW.

Table 3.1: Stratigraphy of Raniganj Field (after Ghosh, 2002)

Geologic Age (m.y.)	Formation
Tertiary	Bangal basin clay, sand and limestone
unconformity.....
Jurassic –Cretaceous	Rajmahal Trap Intratrappeans
unconformity.....
Late	Supra- Panchet
TriassicAngular unconformity.....
Early	Panchet
	-----unconformity (local).....
Late	Raniganj
	Damoda
Permian	group
	Barren Measures
	Barakar
Early	Talchir
unconformity.....
Precambrian	Metamorphic granite gneiss, schists with pegmatite ,metadolerite, dolerite, lymphyre

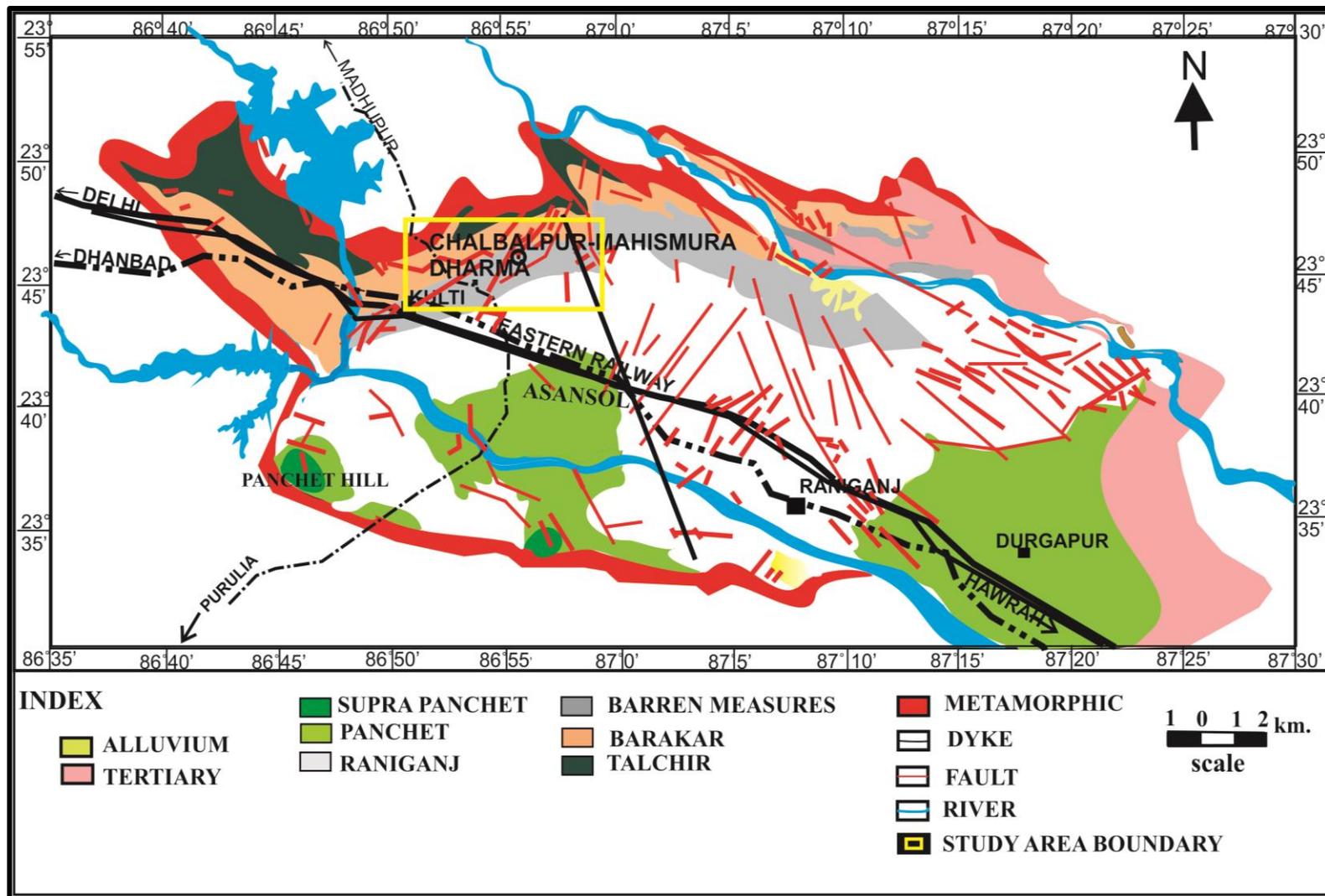


Figure 3.1 Geological map of Raniganj Field (modified after Das, 1992; CMPDIL, 1993)

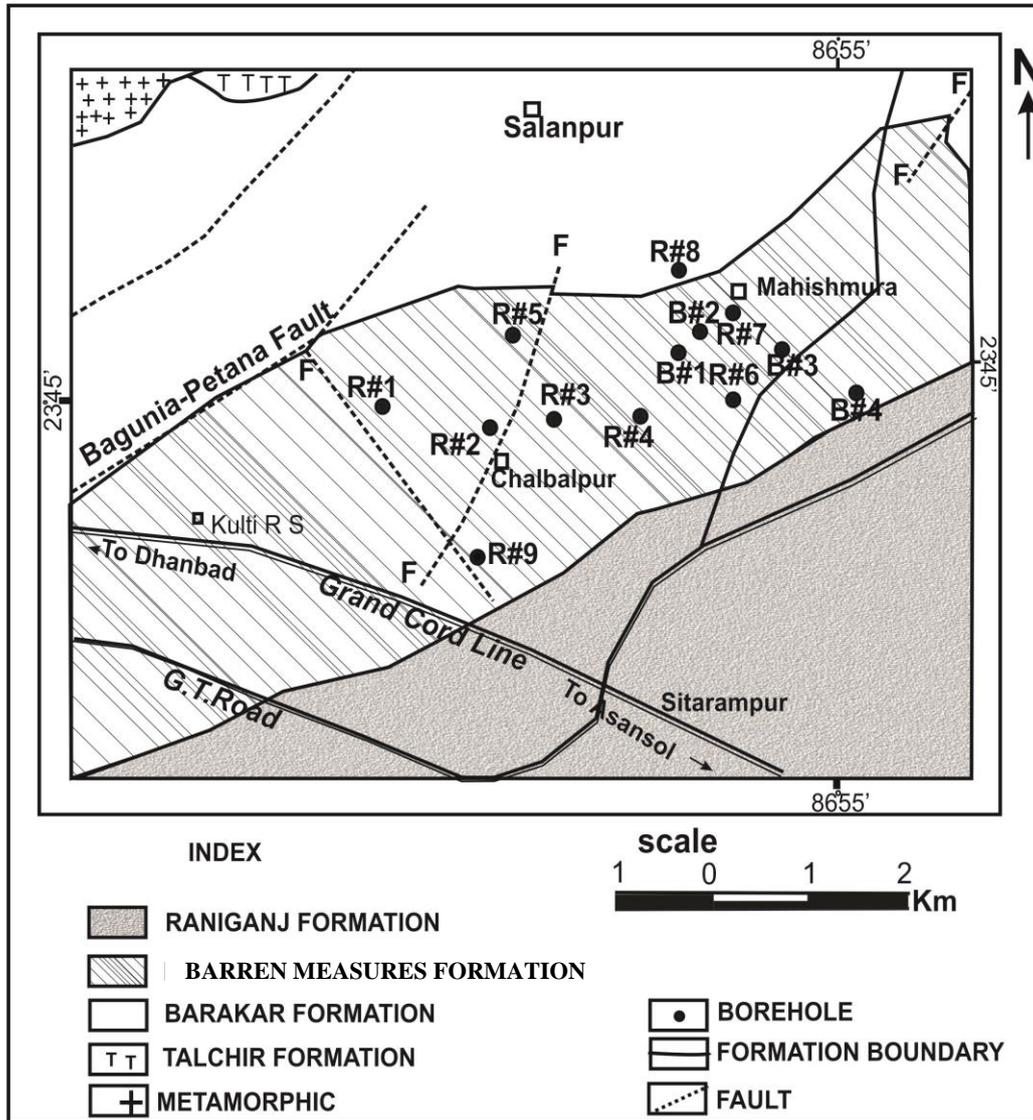


Figure 3.2 Geological map of Chalbalpur- Mahishmura area showing the borehole locations (modified after Das, 1992).

The general strikes of these faults are NE- SW and NW - SE (Chakraborty *et al*, 2003). Dolerite dykes (NNW-SSE trend) runs in the eastern margin of the study area. In subsurface, data reveals few lamprophyre dykes (NE-SW) and sills within the Gondwana formations. The lamprophyre sills intrude into the all formations, while within the study area they intrude into only Barakar Formation. Lamprophyres produced pyrolitized coal which are named locally as ‘‘jhama’’ (Das, 1992).

The study area is located at the NNW part of Raniganj field at Chalbapur- Mahishmura region (Figure. 3.2). The structural set-up of the Chalbapur-Mahishmura area is controlled by regional geological settings. The sediments in this area have a regional low dip towards south. The dip angle varies from 3° to 7°, which indicates the local tectonic disturbance of the strata. The Begunia-Petana strike fault is trending ENE-WSW for almost 3.5 kilometers in the study area, which separated the Barakar and Barren Measures Formations on the surface. The sediments encountered in the boreholes (R#1, R#2, R#3, R#4, R#5, R#6, R#7, R#9, B#1, B#2, B#3 and B#4) of the area, belong to the Talchir, Barakar and Barren Measures Formation. All the boreholes (except R#8) were drilled through a thick sequence of Barren Measures Formation for exploring the Barakar seams occurring below. But the borehole R#8 location is directly rest on Barakar Formation. On the basis of borehole data of R#2 & R#5, the NNE-SSW and NW-SE trending cross-faults (F1 & F2) have been observed. However, no surface expression is seen as the area is covered by thick alluvium of 15m to 18m. Besides, a number of minor faults have been recorded in the boreholes.

Basement [Age: Pre- Cambrian]

The rocks of Pre-Cambrian basement are exposed on the flanks of the Raniganj graben, and are occurring in the NW part of the study area and comprise gneiss, schists with pegmatite and intrusive of metadolerite, dolerite, lymprophyre.

Talchir Formation [Age: Late Carboniferous to Early Permian]

The Talchir rocks are exposed towards NNW part of the study area, resting unconformably over the metamorphic basement. The Talchir Formation was encountered in borehole R#1, R#5 & R#7 having thickness of 10m, 12m and 93m respectively; reflecting the thickness increases towards east within the study region. The rocks of the Talchir comprise fine grained, dirty white to green coloured sandstone, siltstone with black to greenish black silty shale and basal conglomerate. The silty shale becomes splintery in nature, when exposed to

atmosphere. The characteristic of the rock units represent glacial and glacio fluvial environment (Chakraborty *et al*, 2003). Out of thirteen boreholes, R#1, R#5 and R#7 have penetrated into the basement metamorphic, while rest of the boreholes was closed within the Barakar Formation.

Barakar Formation [Age: Early Permian]

The Barakar Formation is unconformably overlying the Talchir Formation and comprised of sandstone, shale, carbonaceous shale and coal seams (Figure 3.3) whereas towards the top, gradational contact with Barren Measures are observed. The sandstones are fine to coarse grained, sub-arkosic and poor to moderately sort. The numbers of regional coal seams are encountered in boreholes within the Barakar, and have named by Coal India Ltd and Geological Survey of India in descending order, are Begunia, Ramhagar, Laikdih, Salanpur 'D', Salanpur 'C', Salanpur 'B' and Salanpur 'A'.

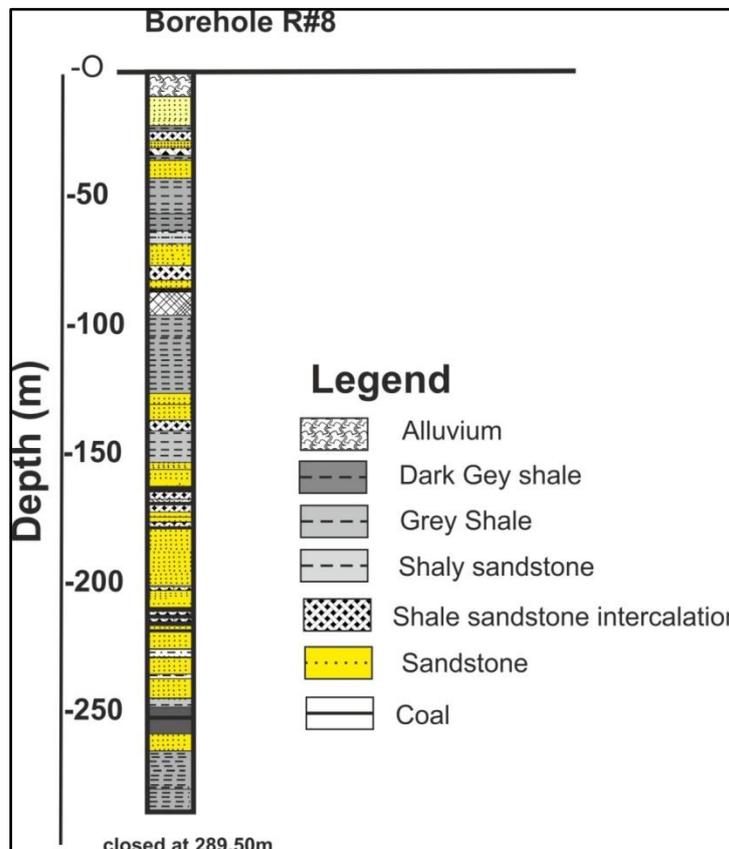


Figure 3.3 Barakar lithology at borehole R#8

Within the study area, the average thickness of the Barakar strata has been measured from the available borehole data is 492.68 m. Detailed study of the sub-surface data reveal both vertical and lateral variation in the lithofacies. The sediments are characterized by the dominance of fine to medium grained sandstone with sandstone/shale intercalation and shale. Three to four layers of very thick (25 m- 35 m) and continuous shale horizons have been recorded in this formation. The ultrabasic intrusive are prolific in this part of Raniganj Coal field and most of the higher quality coal seams like Begunia, Ramnagar and Laikdih seams are totally or partly affected by these intrusives. The lower part of formation is represented dominantly by coarse to very coarse grained sandstone with frequent occurrences of granular to pebbly horizons, sandstone/shale intercalation and shale and coal seams. The sediments show fining upward cycles. The lithology of R#8 is presented here, the other logs are given along with Barren Measures Formation.

Barren Measures Formation [Age: Late Permian]

The objective and target of this work is to understand the geology of the Barren Measures Formation, its lithofacies variation, mineral composition, diagenesis, petrophysical characters etc. The detailed field, borehole litholog study and laboratory investigations have brought out immense data which are presented in the proceeding paras.

Field Investigation and Borehole Litholog Analysis

The Barren Measures Formation is overlying the Barakar Formation with a gradational contact. The contact of Barakar Barren Measures in different boreholes represents sandstone/shale (in R#9), sandstone shale intercalation (in R#6,R#4,R#3) and sandstone/sandy shale (in R#2, R#1). The Barren Measures are distinguished from Barakar as they contain Fe-claystone band and lack of coal seams. The exposures were seen along nala section at Chalbalpur- Mahishmura area. The contact between Barren Measures and Barakar Formation is exposed along Nunia nala (rivulet). The red line in Plate 3.1 demarks the contact

and this measured exposed section (latitude N 23°45'23.59'', longitude E 86°54'22.57'') is having 10 to 15 feet cliff. A generalized litholog of vertical section is presented in figure 3.4.



Plate 3.1 The Barren Measures exposes along Nunia nala section. The red line indicates the Barren Measures and Barakar contact.

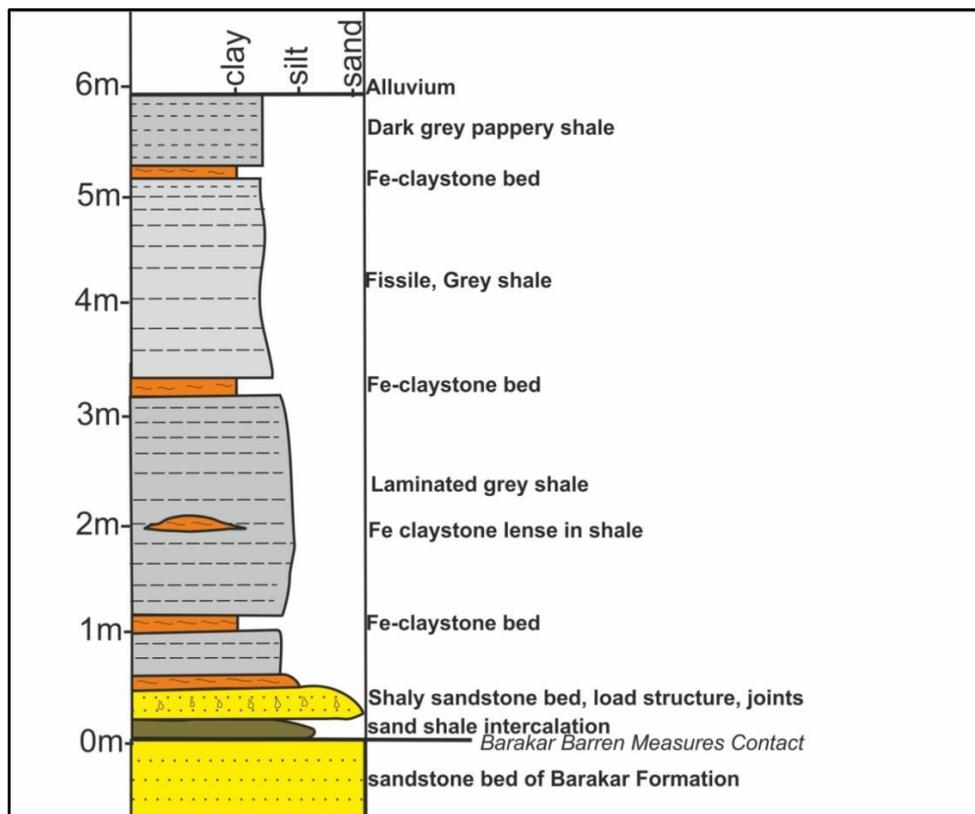


Figure 3.4 A generalized litho column of the cliff section (Location: Nunia Nala).

The Barren Measures Formation comprises grey to dark grey shale with presence of thin Fe rich claystone bed (0.15 to 0.20m) and sand intercalation at the base (Plate 3.2 A, C). The Barakar Barren Measures contact is distinguished by the intercalation of thin sand and shale layers. The exposure of the Barren Measures rocks is limited, are eroded towards downstream of the nala, where only a sandstone bed of Barakar Formation is exposed approximately 400 m along the nala.

The Barren Measures shale is grey to black in colour, laminated, fissile to papery, indurated, soft, brittle, flaky and occasionally brownish grey in colour. The fissility of shale is observed to be increases upwards. The clay stone bed is hard, compacted, blocky and reddish brown in weathered condition while the fresh surface is grey (Plate 3.2 A). The shales are occasionally altered into yellowish green due to weathering effect (Plate 3.2B). The regional strike of the bed is ENE-WSW with 5° to 7° southernly dip. Near Mahishmura village the amount of dip slightly increases to 10° - 15°. Conspicuous joints have been observed in sandstone units, where the trends are NW-SE (J1) & N-S (J2). The J2 joint set is spaced more closely towards east. Often shales also exhibit joints (Plate 3.2 C, D) but there is no clear directional trend; this could be on account of their brittle and ductile characters. The presence of natural fractures is demarcated by red dotted line in Plate. 3.2 F. The basal unit of Barren Measures exhibits load structures (Plate 3.2E); representing the soft-sediment deformation structures, developed during deposition or first stages of the sediment's consolidation but prior to cementation under high sediment deposition rates. The structures were deformed due to water escape. The bed load structures may result due to the application of external load (e.g. soft sediment folding/faulting) or due to density instability between different sediments layers. These irregular bulbous features formed as a denser material has sunk into a less dense material or denser material pinches off (Bogg, 1987).

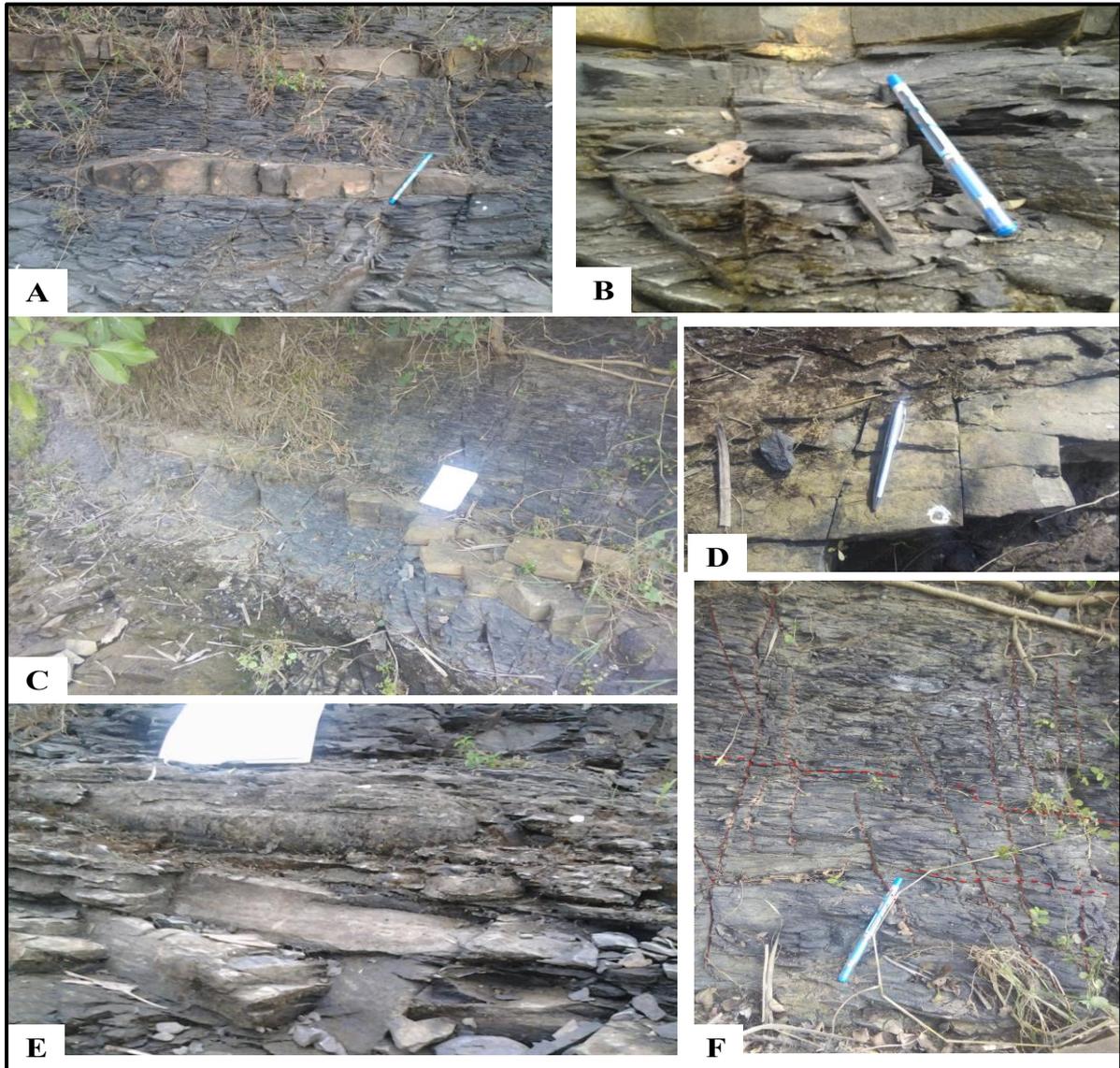


Plate 3.2 *Lithofacies and structures in Barren Measures Formation (Location: Nunia Nala)*

- A. Ferruginous claystone bed and lenses within grey shale,*
- B. Fissile grey shale. Colour alteration from grey to green due to weathering effect*
- C. Thin bed of massive Fe rich claystone (15 to 20cm), interbedded with fractured shale,*
- D. 2 sets of joints on the rock surface*
- E. Shale sand intercalation, bed load structure at the base of Barren Measures shale;*
- F. Natural fractures on the shale rock surface, fractures are along the bedding plane, perpendicular to bedding and cross cutting fractures.*

Thicknesses of the Barren Measures Formation, as intercepted in the boreholes, have been tabulated in table 3.1, where the thickness ranges from 80m to 339m. Figure 3.5 shows the lithologs of boreholes R#1, R#2, R#3, R#4, R#5, R#6, R#7, R#9. The litho-units of the Barren Measures are presented in Figure 3.6.

Table 3.1 Thickness of different formations as encountered in the borehole (source: Das, 1992).

Borehole Name	Total Depth (m)	Alluvium (m)	Barren Measure (m)	Barakar (m)	Talchir (m)	Metamorphic (m)
R#1	647.80	10.00	80.00	538.00	10.00	9.80
R#2	685.00	15.00	218.00	452.00	Not Drilled	
R#3	847.50	11.00	174.00	662.50		
R#4	836.00	15.00	265.00	556.00		
R#5	857.40	11.00	111.20	442.80	12.00	8.40
R#6	800.95	11.00	299.00	547.40	Not Drilled	
R#7	789.50	11.00	100.00	689.95	93.00	2.95
R#8	289.50	12.00	00.00	277.50	Not Drilled	
R#9	987.75	11.00	311.00	665.75		
B#1	590.00	10.00	215.00	375.00		
B#2	567.00	11.00	198.00	358.00		
B#3	669.00	10.00	170.00	489.00		
B#4	689.00	11.00	339.00	350.00		

For better understanding of subsurface geology of the area, fence diagram was prepared and presented in figure 3.7. It is easy to understand that by and large, Barren Measures rocks are distributed over the region and attitude is sub horizontal or low dipping. The faults which are present in the area are not only disturbed the Barren Measures but also Barakar, Talchir and metamorphic at many places, which is clearly observed from the fence diagram. Apparently the thickness is increasing towards E and SE.

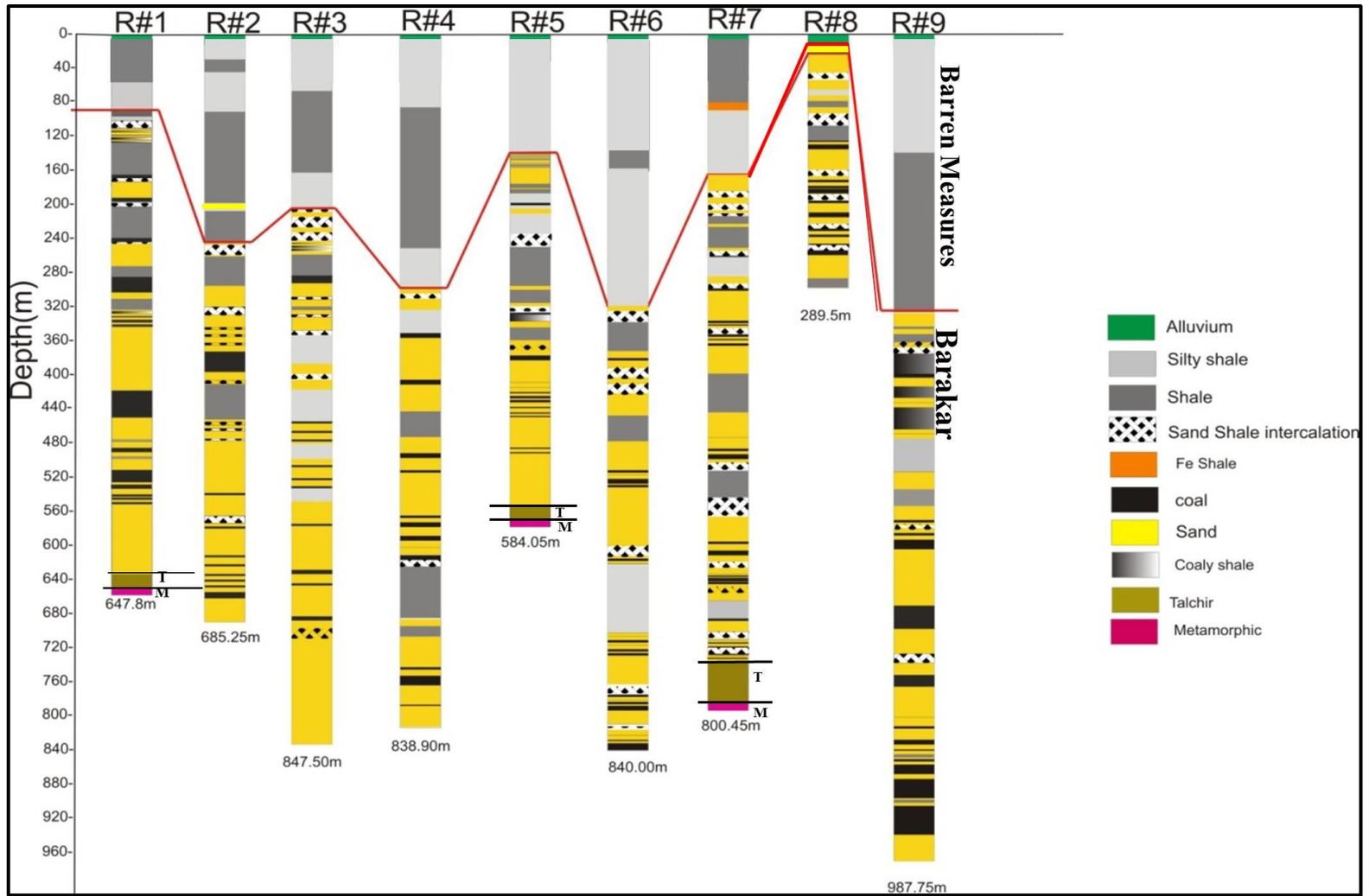


Figure 3.5 Borehole Lithologies showing litho units of Barren Measures and Barakar Formations. Talchir and Metamorphic Basement were encountered in boreholes R#1, R#5 and R#7 while rests of the boreholes were terminated within the Barakar Formation.

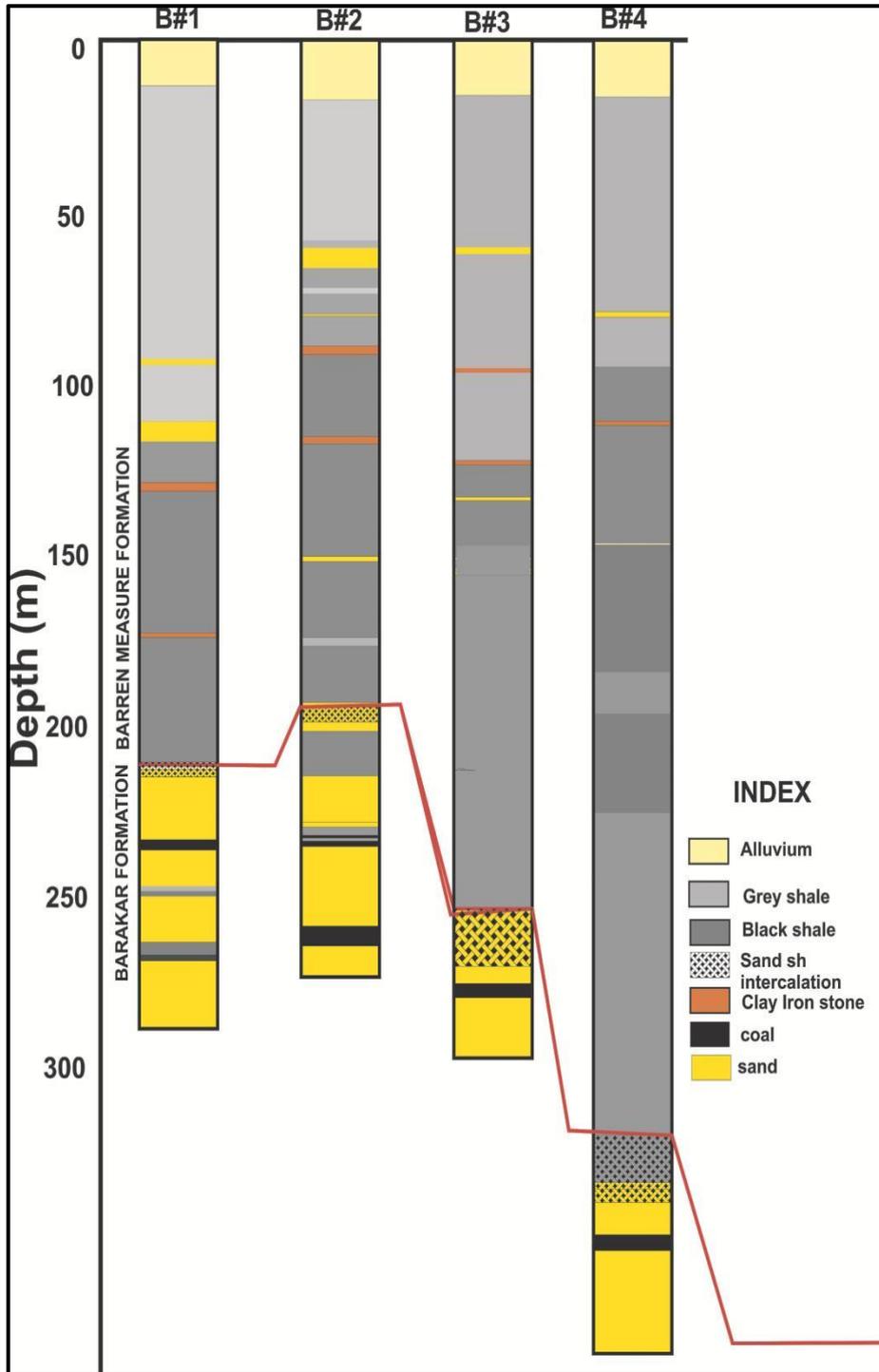


Figure 3.6 Log correlation profile showing the Barren Measures litho units.

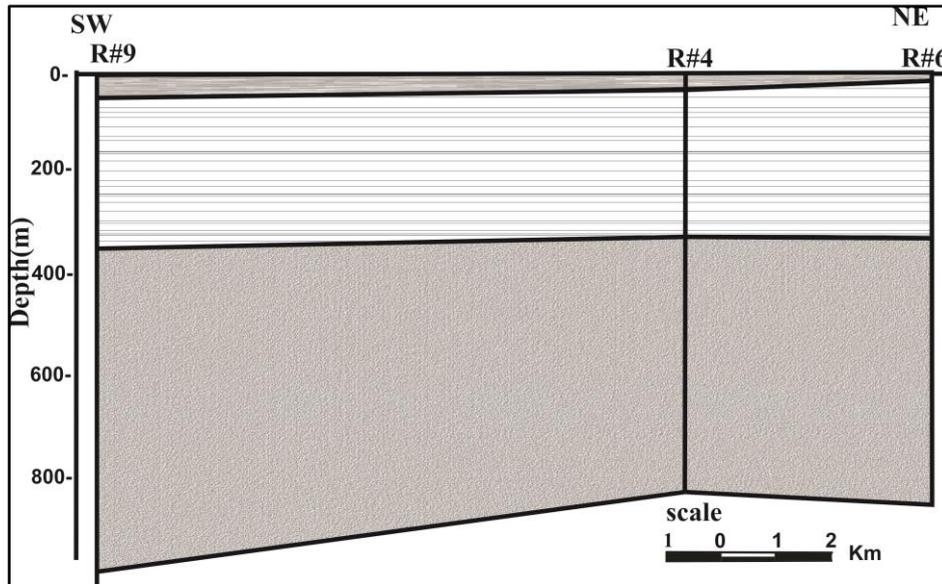


Figure 3.8 B SW- NE trending profile (Profile 2) along R#9, R#4, R#6. The thickness of Barren Measures shale increases towards south direction.

To understand the effect of fault in the study area, cross sections were prepared along borehole R#1, R#2, R#3 and R#4. It reflects the down faulting of the western side block (Figure 3.8A). SW- NE trending profile along R#9, R#4 and R #6 reflects the Barren Measures Formation is having uniform thickness whereas Barakar shows more thickness towards south west (Figure 3.8B). To have a glimpse of the depositional fill in the study area, the structural contour map on the bottom of Barren Measures (top of Barakar) was prepared and shown in Figure 3.9. The structure contour map also reflects the effect of faults. In western margin and NNE part, the closeness reflecting steep slopes whereas in the central and SE, larger intervals in the contour map reflect sub horizontal nature. The structure contour at the top of Barren Measures was not constructed on account of the absence of Raniganj Formation in all boreholes which were considered in this study. The thickness of alluvium marks the top of Barren Measures. To appraisal the volume of Barren Measures, isopach map was prepared by using thickness of Barren Measures from the borehole data (Figure 3.10). The thickness of the Barren Measures was considered by taking the deduction thickness of alluvium. Raniganj Formation is not recorded in any of the boreholes.

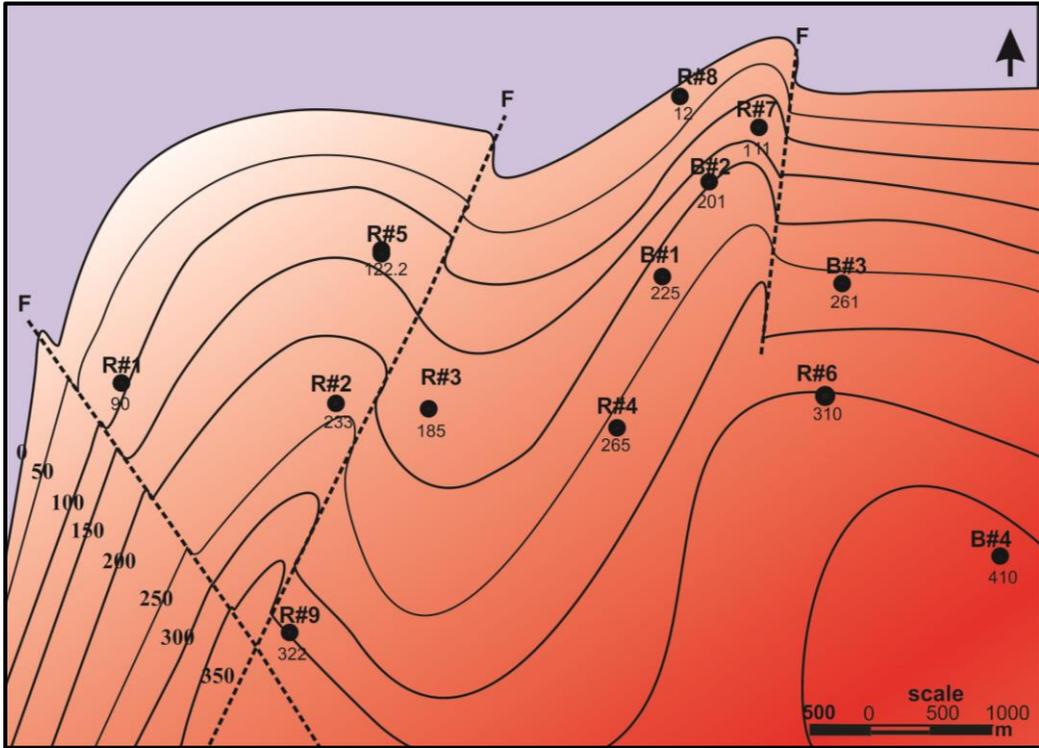


Figure 3.9 Structure contour map on the bottom of Barren Measures Formation.

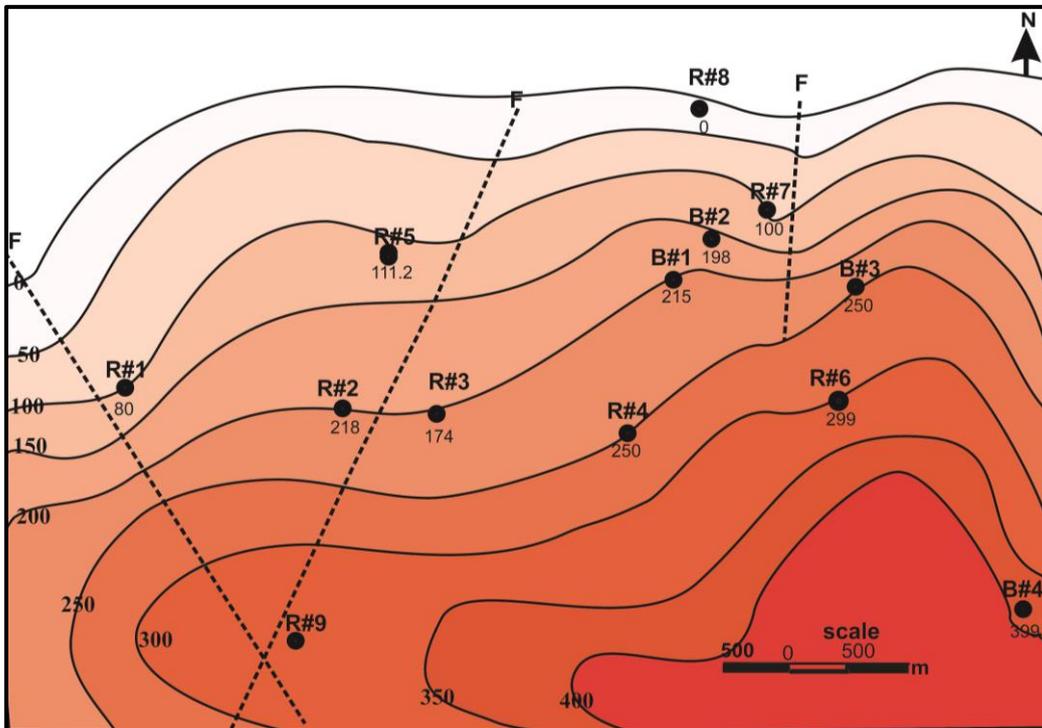


Figure 3.10 Isopach map of Barren Measures Formation

Core Sample Analysis

Core samples were collected from four boreholes i.e. B#1, B#2, B#3 and B#4 and the samples were studied systematically. The megascopic study of the core samples reveals that the Barren Measures Formation exhibits as dark grey to dark black in colour, laminated to blocky, fissile to hard massive (Plate 3.3 A-D). The silt size quartz (>45%) and mica (5 to 10%) grains are present in large quantity (almost 5 to 10%). The rocks are tight, compacted and pores are rarely visible. The lower part of the formation is composed of thin layers of fine to medium grain sandstone and shale intercalation. Thick sequence of carbonaceous and siliceous shale is the main composition of this formation. Fine to medium grain sandstone are present as thin laminae or sometimes as small irregular patches; moreover, ferruginous lamina are very common. Impression of woods and plant leaves were found frequently in cores of boreholes (Plate 3.4 A and B). Often the patches of siderites are occurring on the rock surface (Plate 3.4C). Fracture filling organic matters (Plate 3.4 D) and 2 sets of joints (cleats like structure) are observed in the core plugs (Plate 3.4E)

Based on megascopic and microscopic studies of Barren Measures shales, the litho units are recorded as:

- a) Dark grey to black carbonaceous fissile to papery shale
- b) Grey to dark grey carbonaceous silty shale
- c) Fe rich claystone
- d) Shale and sand intercalation

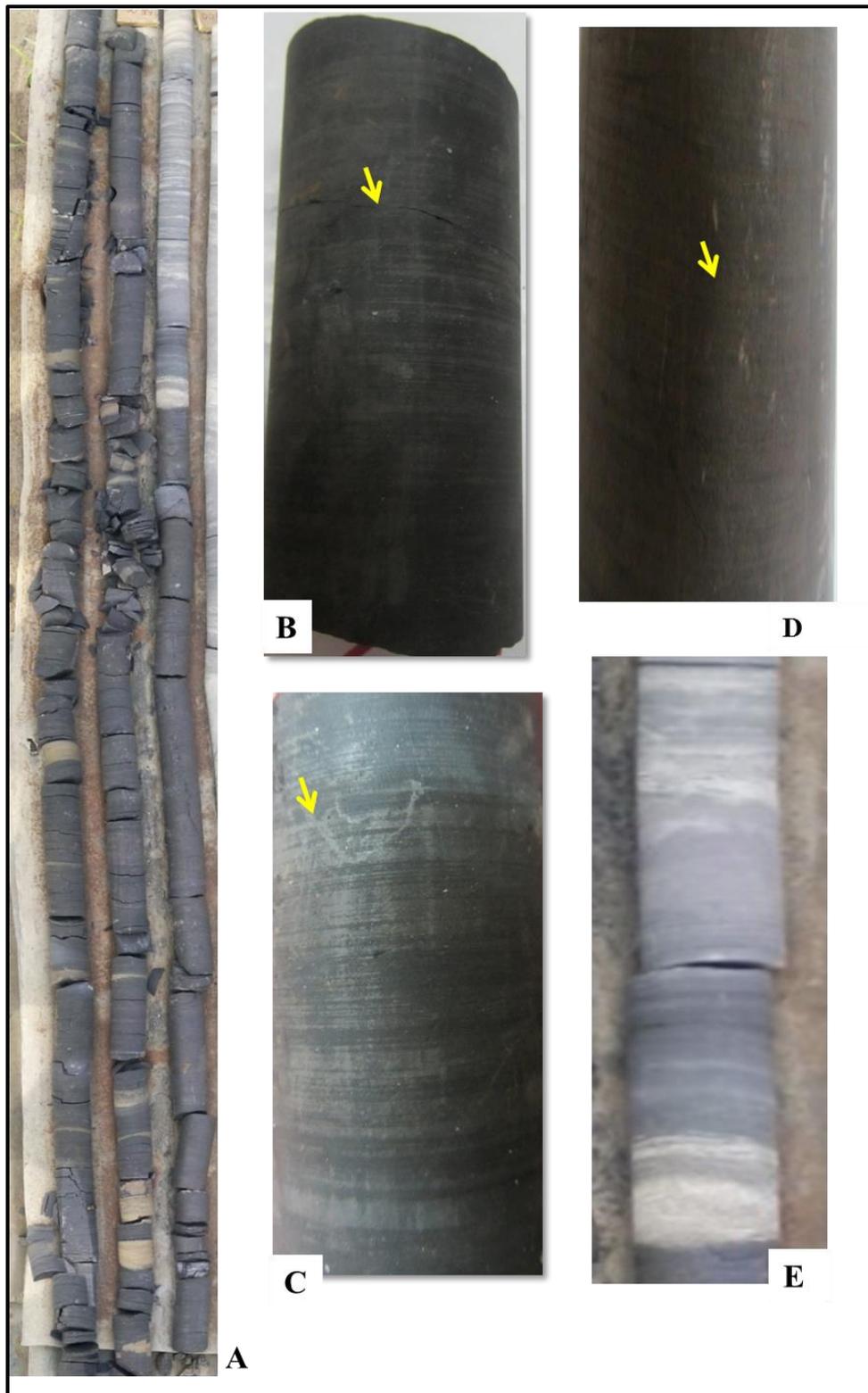


Plate 3.3 Core samples of Barren Measures shales; Borehole B#1. A. cores of Barren Measures Formation; B. Laminated blackish grey shale, C. Laminated grey shale, colour differentiation due to mineralogy or presence of organic matter; D. Fe-claystone; E. Sand shale Intercalation.

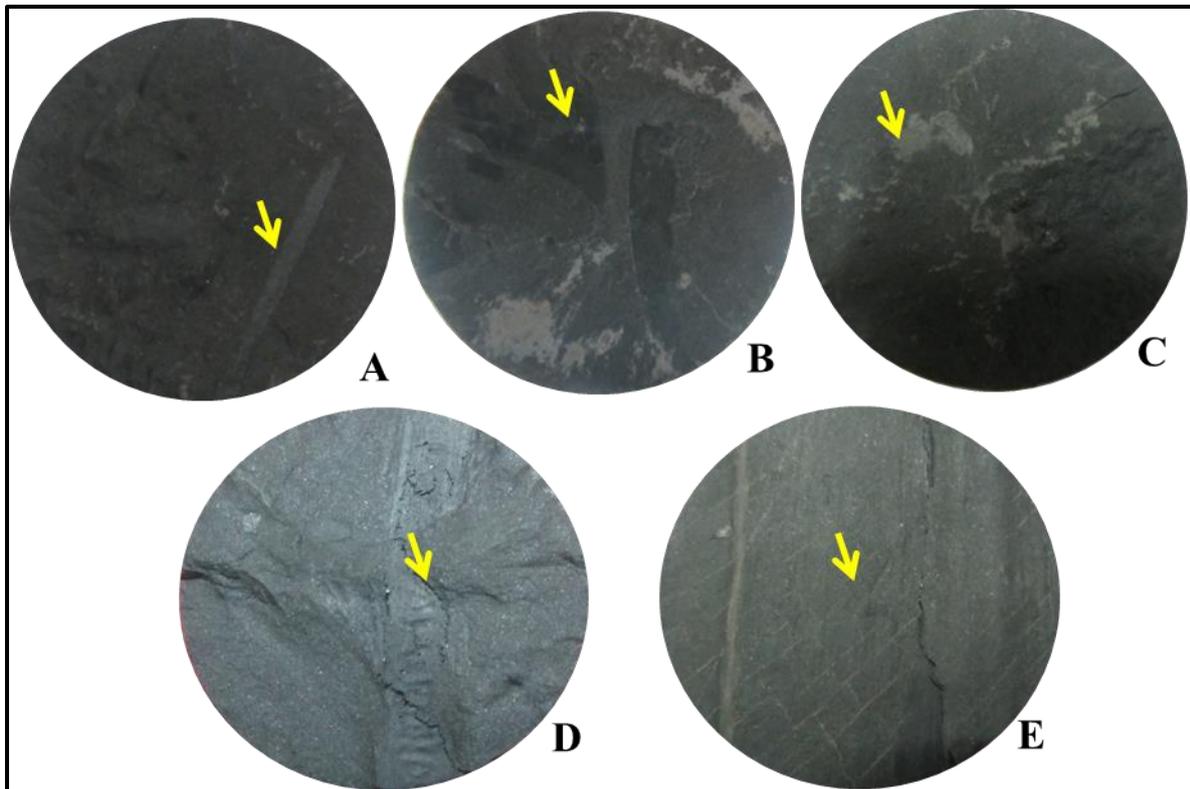


Plate 3.4 Core samples of Barren Measures shales in cross section view; Borehole B#1. A & B. Plant leaf impressions; C. Patches of siderite; D. Fracture along the plant leaf impression where fracture is filled with fine particles of organic matter; E. Micro fractures

The black carbonaceous shale unit is fissile, brittle with abundant plant leaf impressions on the rock surface. The clay mineral content is high. Organic matters are dispersed on the matrix. Silty shale unit is micaceous and often carbonaceous, dark grey to black, laminated to blocky (massive) in nature, hardness is moderate. The laminations in the shales are due to presence of thin layers of silt and clay, and sometimes colour alteration from grey to black due to presence of organic matter in differential quantity. The presence of laminated to blocky shale units reflects both uniform and differential rate of sedimentation. The weak plan is parallel to the bedding. The iron rich shale unit is dark grey in colour with frequent iron lamination of brownish colour. This unit is very hard, tight and well compacted.

Petrographic Analysis

The thin sections of samples (n=50) were studied using standard petrographic techniques (Greensmith, 2012) with the help of polarizing microscope to envisage their petrographic

characters such as mineral constituent, porosity, textures, fabrics, diagenesis etc. (Potter *et al*, 1980; Pettijohn, 1984; Adam and Mackenzie, 1998). Under microscope, the Barren Measures shales were observed to be composed of quartz, lithic fragment and iron rich, carbonaceous, argillaceous matrix. Quartz is the most abundant grain type in Barren Measures shales (Plate 3.5 and 3.6). Microscopic observation shows alternate silt and clay laminations (Plate 3.5A, 3.5C, 3.5E and 3.6F) whereas organic matter flakes are abundant in the black carbonaceous shales. Plate 3.5B shows the composite quartz grains under crossed polars, where the crystals are elongated in a preferred direction and each individual crystal is showing undulose extinction, may be due to strain grains derived from the stretched quartz of metamorphic origin. Mica, mainly muscovite is forming more than 10% of the rock (Plate 3.5F, 3.6D). The source of muscovite is generally a granitic or schistose rock. The parallel alignment of the quartz grains and muscovite flakes indicates the bedding (Plate 3.5E, 3.6 C 3.6 D, and 3.6 F). Concentric iron nodules of ~200 μ diameter were seen (Plate. 3.6A) were seen under microscope. These are may be iron-bearing siderite nodules, formed due to diagenesis effect. Concentric layering in iron-bearing siderite nodules is thought to effect from agitation associated with currents (Plate 3.6B). Fine grain quartz dominant, iron oxide and feldspar are common in ironstone shale (Plate 3.5D). Iron flakes are also seen (Figure 3.5D). The presence of sub rounded to well-rounded quartz grain in the shale units indicates a long distance of transport and/or polycyclic nature. The assemblage of quartz + sericite + siderite and/or pyrite, refers phylomorphic alteration at high temperatures and moderately acidic (low pH) and moderate Eh conditions (Pettijohn 1984). The compaction effect is evidenced by point, straight and concavo-convex contacts of framework grains. Compaction has resulted in a reduction of the original size of pore bodies and pore throats, and therefore has contributed to a general reduction in porosity and permeability during burial. However, the floating grain contact is seen (Plate 3.5 B) in most of the studied samples which indicates the

possibility of intergranular pores. In most of the samples, the intergranular spaces are filled (fully and/or partially) filled with organic matter (Plate 3.5 F)

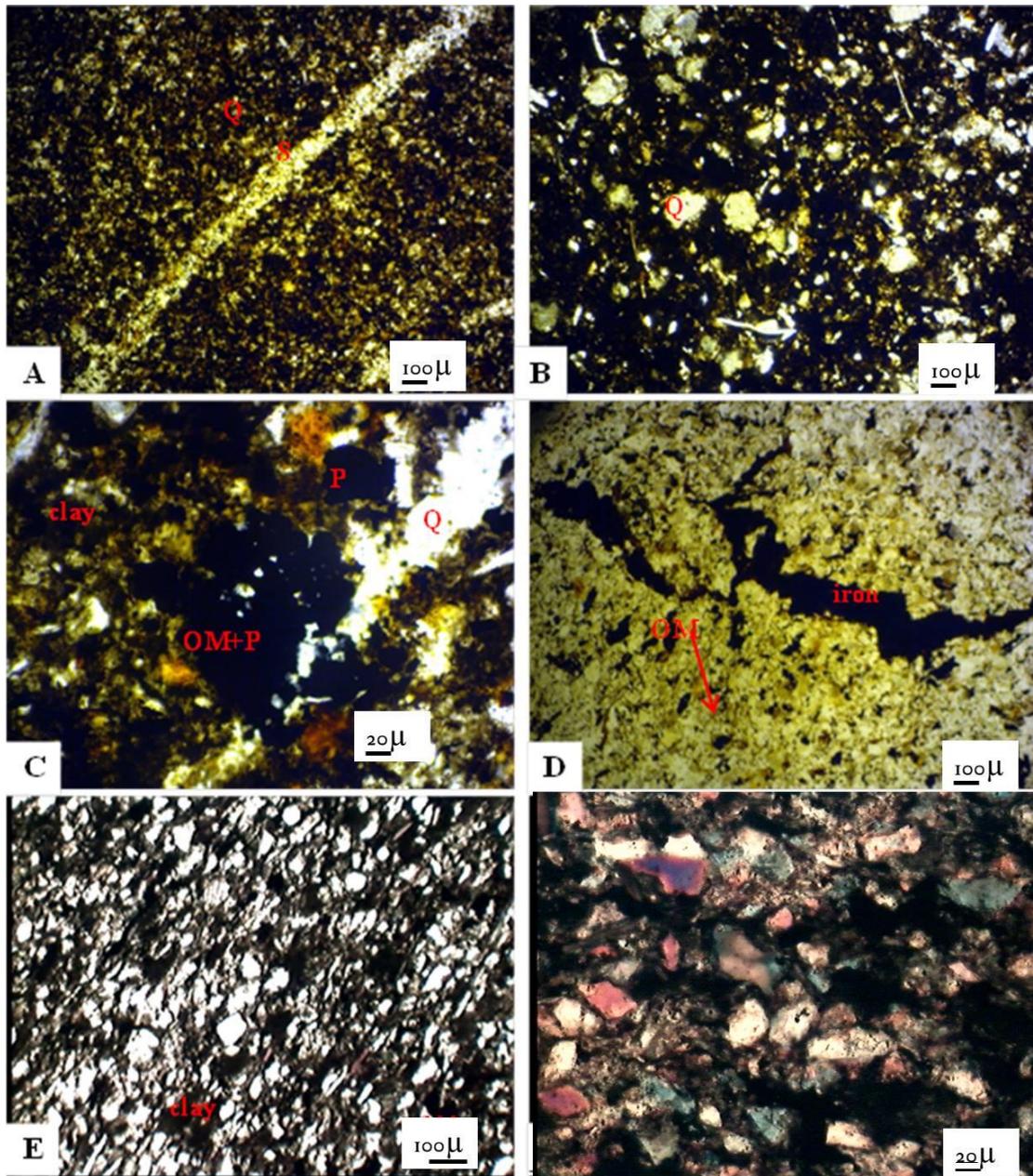


Plate 3.5: Photomicrographs of Barren Measures shale (OM=organic matter, P=pyrite, Q=quartz, S=sericite). **A.** Quartz filled fracture shows altered sericite (Rock type: Ironstone shale, Depth 157m, B#3). **B.** Floating grains of moderately sorted quartz. (Rock type: Silty shale; Depth 125m, B#3). **C.** Silica is coated with pyrites (Rock: Carbonaceous silty shale; Depth 170m, B#1). **D.** Iron flakes coated with organic matter. (Rock type: Ironstone. Depth 105m, B#2). **E.** Quartz and mica grains show parallel orientation forming lamination. (Rock type: Carbonaceous shale; Depth 155m, B#1). **F.** Intergranular spaces are filled with organic matter and clays (Rock type: Carbonaceous silty shale; Depth 145m-150m, B#1).

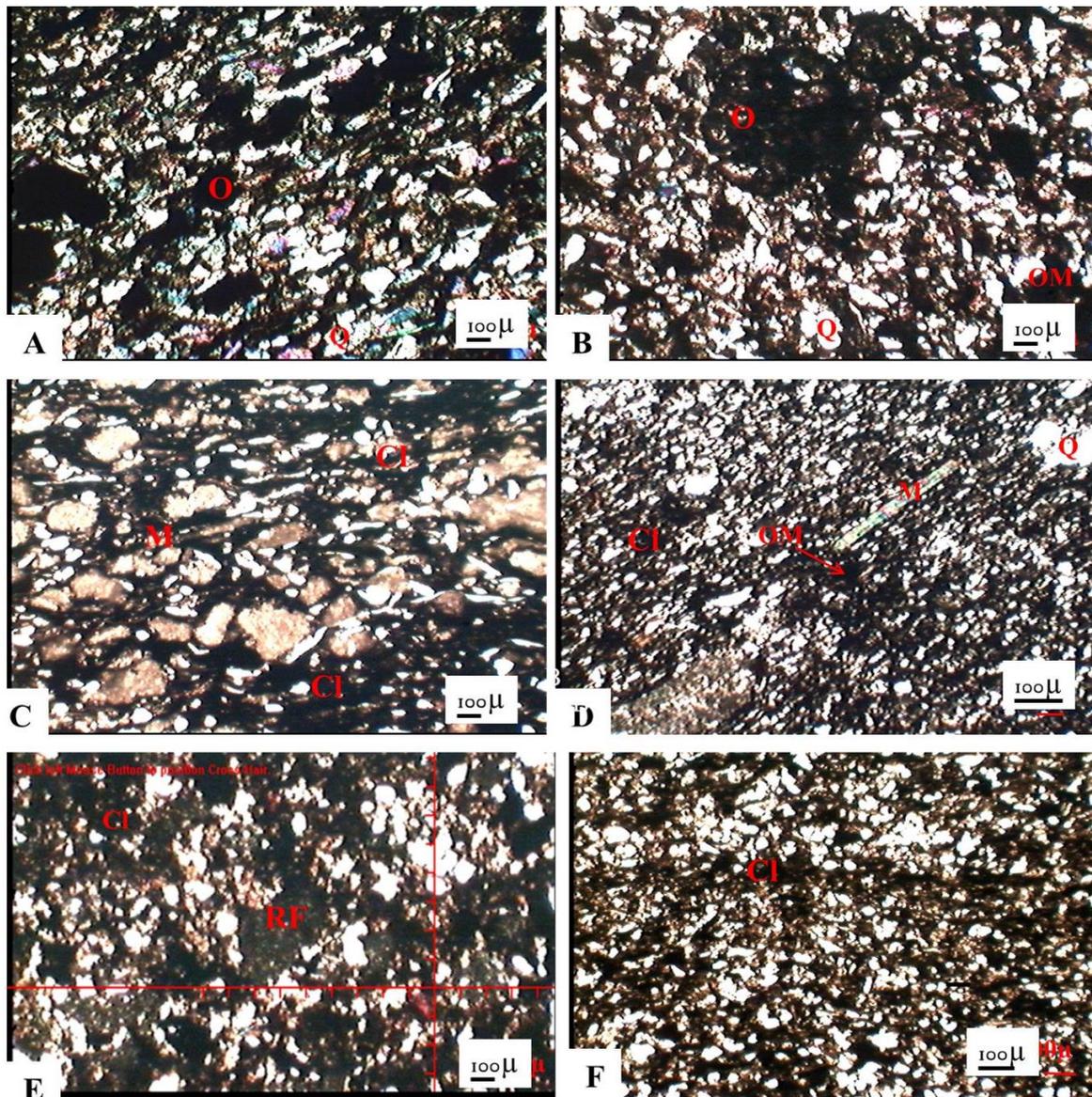


Plate 3.6 Photomicrographs of Barren Measure shale; **A.** Silty shale, fine grain quartz, feldspar, lithic fragments are present along with iron nodules. Organic matter and pore are seen; reflect phylomorphic diagenesis; (Rock type: Ironstone shale; depth 91-93m, borehole B#2). **B.** Fe nodules (Rock type: Ironstone shale; depth 96m, B#2). **C.** Sub rounded, moderately sorted quartz grains are floating on the argillaceous micaceous matrix. Mica grains are lying parallel. (Rock type: sideritic shale; depth interval 125m-130m, B#2). **D.** Mica phenocryst. Quartz grains are of silt size, well sorted, laying parallel, silt and mica grains are align parallel forming lineation. Pore filling organic matters are present; (depth 160m, B#2). **E.** Lithic grains are embedded in clayey matrix (Rock type: claystone; depth 135, B#3). **F.** Clay lamina (depth interval 155m-160m, B#1).

Mineral Composition analysis

The mineralogical composition of shales was determined by X-ray powder diffraction analysis. It includes determinations of the bulk (whole rock) composition of shales and relative abundance of clay minerals. In all 32 core samples from four boreholes were analysed and their results are given in the table 3.2 to 3.5. The XRD result reflects silica dominant minerals occur in all samples from all depth, followed by sulphate of iron, feldspars and micas. Alumino silicates (Andalusite, sillimanite) pointing to metamorphic source of provenance which is also supported by pyrolusite, pyrrhotite. The ilmenite and beryl along with fresh feldspars indicates the acidic igneous rocks as the source. In B#1, at a depth of 170m and 180m, barite mineral is recorded, which suggest reducing condition during time of deposition (Randolph *et al*, 2003). However, barite is not recorded in any other samples. Sulphate-sulphide mineral suggest negative to slightly negative Eh condition. The clay minerals such as chlorite and its family represent their stage of diagenesis is phyllosomorph (Potter *et al*, 1980), suggesting upliftment at the later period. Other clay minerals mainly dominated by illite, smectite, montmorillonite, etc. points to more alkali condition i.e. high pH Condition during deposition. Kaolinite clays represent generally near surface sediment deposition with low pH condition. It indicates the alteration of fresh feldspar into kaolinite under low/neutral pH condition. The feldspar mineral indicates the granitic source of sediments.

The XRD analysis results the average mineral composition of Barren Measures shale as quartz (49.02%), albite (7.07%), pyrite (1.12%), pyrolusite (2.27%), muscovite (1.52%). Pyrrhotite (2.22%) and siderite (5.14%) have been recorded. Whereas the clay minerals are represented by clinocllore Ferroan 13.11%, illite 5.02%, and kaolinite 1.20%. The XRD derived Barren Measures shale mineralogy data are summarized in Table 3.2 to 3.5. The results are represented as bar diagram for easy interpretation (Figure 3.11 to 3.14).

Table 3.2: XRD results of Borehole B#1

Depth(m)	ALB	AND	BRT	BERL	CHLT	C-M	CHRD	CLINO	ILT	I-M	ILME	KAO	LUC	MCRO	MON	MUS	PYRT	PYRL	PYRH	QTZ	SIDRT	SILLI	S-K
141-145	10.59	2.53	0.00	0.00	1.70	2.04	0.00	8.50	1.85	0.00	0.00	3.11	0.00	1.51	1.99	3.16	0.00	4.28	0.00	45.60	6.95	1.65	1.65
150-155	5.18	0.00	0.00	1.86	0.00	0.00	0.00	7.41	8.59	0.00	1.32	0.00	0.95	0.00	0.00	1.36	0.00	3.32	3.27	51.60	0.00	1.05	1.05
160-165	6.10	0.00	0.00	1.93	0.00	0.00	0.00	10.75	4.53	0.00	0.00	3.80	0.00	1.57	0.00	2.54	2.96	2.48	2.17	43.90	9.27	0.00	0.00
165-170	4.87	0.00	0.00	0.00	0.00	0.00	0.00	12.22	2.27	3.70	0.00	0.00	0.00	0.00	0.00	0.00	6.63	6.37	0.00	36.10	10.00	0.00	0.00
170-175	5.97	0.00	2.20	2.01	0.00	0.00	0.00	13.63	6.06	0.00	0.00	0.00	0.00	4.43	1.65	3.08	0.00	3.52	0.00	44.50	0.00	1.57	1.57
175-180	4.15	2.16	0.00	3.57	0.00	2.40	0.00	14.05	9.78	0.00	0.00	0.00	0.00	0.00	0.00	0.00	2.94	3.28	0.00	45.90	9.25	0.00	0.00
180-185	5.71	0.00	1.92	0.00	0.00	0.00	0.00	9.61	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	4.88	3.10	2.76	46.00	11.90	2.46	2.46
185-190	12.64	0.00	0.00	0.00	0.00	0.00	0.00	13.01	5.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	3.13	3.17	50.00	3.50	0.00	0.00
190-195	7.65	1.83	0.00	1.38	0.00	0.00	0.00	14.52	2.76	0.00	1.42	0.00	0.00	1.94	0.00	0.00	0.00	2.99	1.53	46.10	5.49	1.75	1.75
195-200	7.90	1.41	0.00	3.42	1.40	1.65	3.16	16.29	0.00	0.00	0.00	0.00	0.00	6.81	0.00	9.64	1.55	3.19	0.00	33.60	7.47	0.00	0.00

Table 3.3: XRD results of Borehole B#2

Depth(m)	ALB	AND	BERL	C-M	CLINO	ILT	KAO	LUC	MCRO	MUS	PYRT	PYRL	PYRH	QTZ	SIDRT	SILLI	S-K
91-95	4.45	0.00	0.00	0.00	22.21	1.51	0.00	0.00	0.00	0.00	0.98	3.51	0.98	66.00	1.28	0.00	0.00
105-110	17.44	0.00	0.00	0.00	11.79	3.87	0.00	0.00	0.00	2.26	0.00	3.38	0.00	49.80	3.44	1.45	1.45
115-120	13.80	1.52	0.00	2.24	10.03	1.44	1.29	0.00	0.00	0.00	0.00	3.31	1.86	48.80	5.82	0.00	0.00
125-130	4.91	0.00	1.82	0.00	19.02	0.00	3.59	0.00	4.19	0.00	5.03	1.35	3.41	36.00	11.97	1.65	1.65
149-150	4.26	1.25	2.06	1.72	19.06	1.42	1.99	0.00	1.08	5.61	1.11	2.30	3.75	46.90	1.96	0.00	0.00
155-155	5.79	0.00	1.66	1.74	16.67	5.91	0.00	0.00	1.35	0.00	0.00	3.09	3.86	51.50	1.66	1.16	1.16
175-180	4.37	0.00	2.22	0.00	13.33	9.67	5.78	1.56	0.00	4.81	0.00	2.26	4.30	40.40	6.48	0.00	0.00

[ALB= albite, AND= andalusite, BRT=barite, BERL=beryl, CHLT=chlorite, C-M=chlorite-montmorillonite, CHRD=chloritoid, CLINO=clinoclroferroan, ILT=illite, I-M=illite-montmorillonite, ILME=ilmenite, KAO=kaolinite, LUC=leucite, MCRO=microcline, MON=montmorillonite, MUS=muscovite, PYRT=pyrite, PYRL=pyrolusite, PYRH=pyrrhotite, QTZ=quartz, SIDRT=siderite, SILLI=sillimanite, S-K=smectite- kaolinite.]

Table 3.4: XRD results of Borehole B#3

Depth(m)	ALB	BERL	CLINO	ILT	KAO	MUS	PYRT	PYRL	PYRH	QTZ	SIDRT	SILLI
110-115	13.73	0.00	6.31	2.79	2.01	0.00	0.00	3.31	0.00	59.80	4.05	0.00
115-120	5.29	0.00	9.54	4.40	9.28	0.00	0.00	2.92	5.14	53.80	0.00	2.37
120-125	15.22	0.00	4.67	0.00	0.00	0.00	0.00	0.00	4.06	57.80	6.31	0.00
125-130	0.00	0.00	25.00	0.00	0.00	5.25	0.00	0.00	5.91	52.50	0.00	0.00
135-140	4.78	3.00	13.56	12.30	1.50	0.00	0.00	3.36	2.68	46.70	7.89	0.00
147-150	14.32	0.00	7.54	4.06	0.00	0.00	0.00	0.00	0.00	56.50	3.71	3.77
150-155	5.18	0.00	4.01	24.60	0.00	0.00	4.60	0.00	4.53	39.30	11.65	0.00
157-160	5.02	0.00	17.94	5.64	0.00	0.00	0.00	0.00	3.70	57.10	0.00	2.67

Table 3.5: XRD results of Borehole B#4

Depth(m)	ALB	BERL	CLINO	ILT	KAO	MUS	PYRT	PYRL	PYRH	QTZ	SIDRT	SILLI	S-K
291-295	5.58	0.00	11.16	4.60	0.00	0.00	0.00	0.00	0.00	56.60	8.77	4.05	0.00
295-297	7.24	0.00	8.88	5.84	2.02	0.00	0.00	0.00	0.00	58.30	6.39	0.00	0.00
301-305	0.00	0.00	19.30	3.53	1.80	4.65	4.20	0.00	5.60	44.10	0.00	4.03	4.03
305-310	4.66	2.92	13.20	14.64	0.00	0.00	0.00	3.28	2.61	45.60	7.68	0.00	0.00
315-320	8.35	0.00	8.99	2.61	1.10	4.86	0.00	4.86	0.00	53.40	3.58	0.00	0.00
320-321	3.92	3.23	24.26	5.99	0.00	0.00	0.00	0.00	4.31	55.00	3.23	0.00	0.00

[ALB= albite, BRT=barite, BERL=beryl, CLINO=clinochloreferroan, ILT=illite, ILME=ilmenite, KAO=kaolinite, MUS=muscovite, PYRT=pyrite, PYRL=pyrolusite, PYRH=pyrrhotite, QTZ=quartz, SIDRT=siderite,

SILLI=sillimanite, S-K=smectite- kaolinite

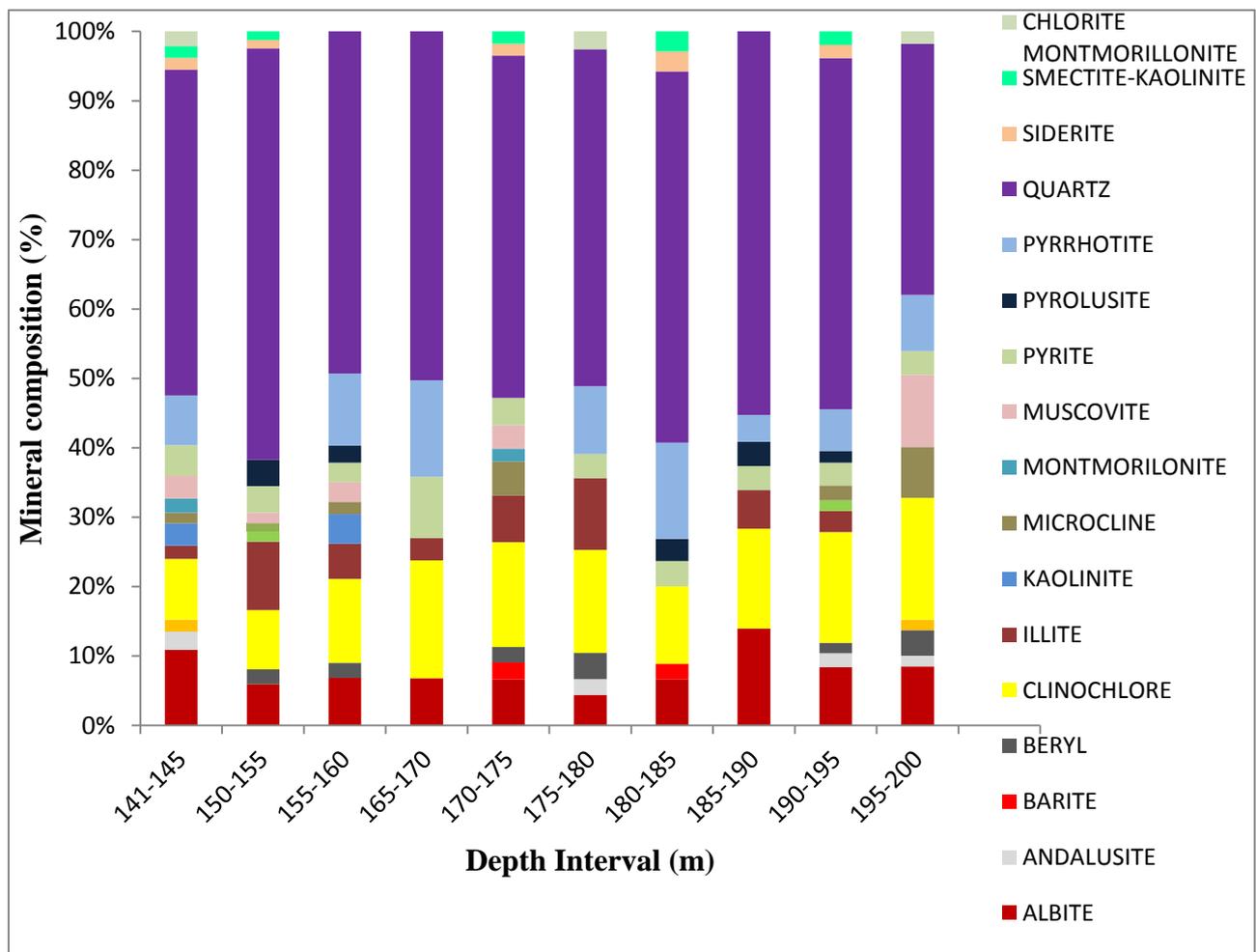


Figure 3.11 Mineral composition of Barren Measure shale for different depth interval of B#1

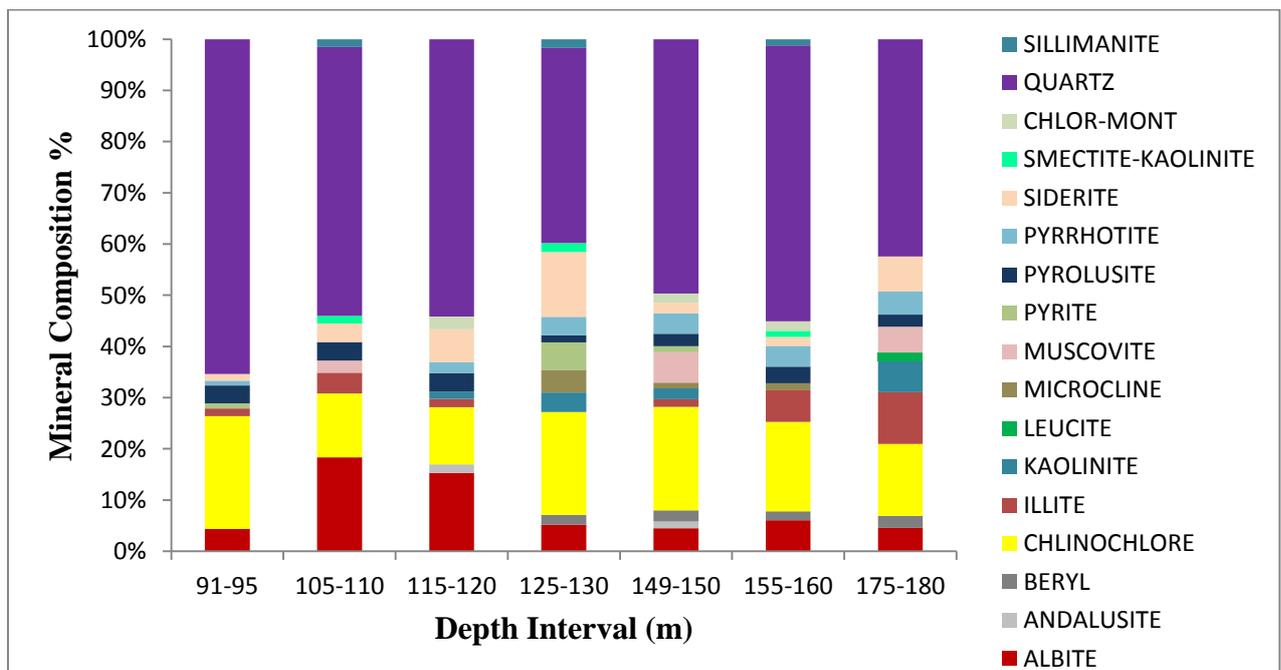


Figure 3.12 Mineral composition of Barren Measure shale for different depth interval of B#2

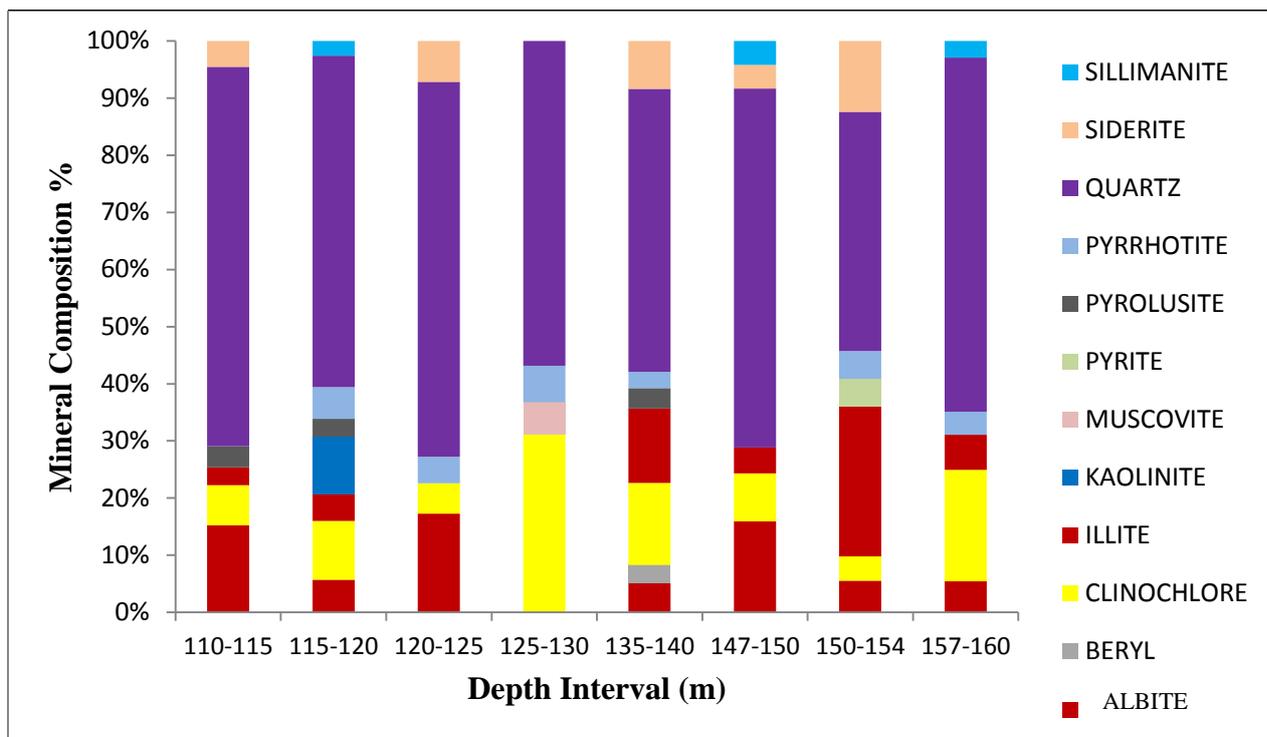


Figure 3.13 Mineral composition of Barren Measure shale for different depth interval of B#3

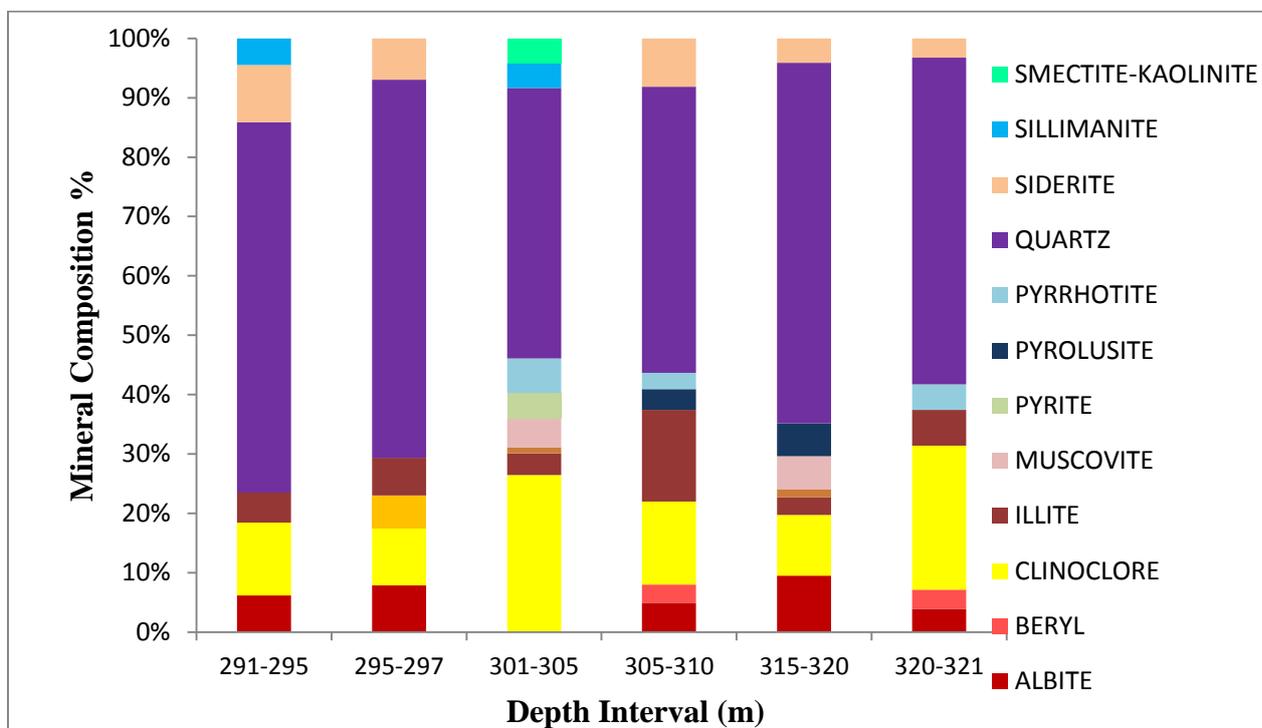


Figure 3.14 Mineral composition of Barren Measure shale for different depth interval of B#4

Thin-section examination and XRD analysis demonstrate that the samples are generally contain less than one-third clay minerals. Detrital quartz and feldspar occur as silt size grain. Organic materials are present in the form of flakes or sub rounded particles and scattered over the rock matrix. The entire Barren Measures Formation is represented by shales with varying colour, grain size and mineralogical assemblages. The changes are gradational, which is reflected in the thin section study and XRD analysis. Vertical changes from one sub units to another are gradational.

a. Iron Minerals

The most common iron mineral are iron carbonates (siderite FeCO_3), iron sulphide (pyrite FeS_2), pyrrhotite ($\text{Fe}_{1-0\text{to}0.2}\text{S}$). The studied shale samples of Barren Measures Formation contain average 1.12% and 5.15% pyrite and siderite mineral respectively. Plate 3.7 indicates the clayey matrix of the shale where mineral grains are coated with clay matrix. The maximum percentage of pyrite is present in the depth interval of 165-170m in B#1 where siderite content is also high (10%). The highest siderite percentage is acquired at the depth interval of 125-130m in B#2. Pyrite is present in isolated (elliptical to spheroid) grains forms. The most common are small grains scattered over the rock surface generally less than 10 mm in diameter (Plate 3.8). Presence of fine grain pyrite framboids/nodules in clay rich shale (Plate 3.8) at depth interval 105m to 110m in B#2 indicates the anoxic condition of sedimentary deposition which suggest high organic content (Shen *et al*, 2007). This may be result of a slow burial of the sediments during the time of deposition in lakes with prevailing anoxic conditions which leads to preservation of carbon and carbon compounds. The occurrence of both pyrite and siderite in the Barren Measures Shale refers that the shale was deposited under an anoxic sedimentary environment where reduced iron (Fe^{2+}) can exist in

solution. The source of iron is the reduction of detrital iron oxides in a strongly reducing, organic-rich sedimentary environment given by:



Sulfides are produced from sulfate ($\text{SO}_4^{=}$) during bacterial decomposition of organic matter (Berner, 1974), Organic decay also provides some sulfate to the sedimentary environment.

Siderite forms through the combined effects of iron reduction and bacterial methanogenesis of organic carbon compounds. Fe^{++} is precipitated as siderite only in "nonsulfidic environment".

According to Pye *et al*, 1990, the Fe reacts with sulphide to form pyrite, so that siderite will not form unless the rate of Fe production exceeds the rate of sulphide production. In short, the pyrite was deposited under reducing condition. When rising at the surface of swamps and marshes, the water becomes oxygenated and siderite was formed.

b. Clay Minerals

Illite is the dominant clay mineral in Barren Measures shales which may be formed by the weathering of silicates (primarily feldspar), through the alteration of other clay minerals, and during the degradation of muscovite. Formation of illite is generally favored by alkaline conditions and by high concentrations of Al and K. The illite in the Barren Measures shales are characterized by intense 10.01-angstrom, 2.34-angstrom peaks.

The presence of mix layers clays i.e. chlorite montmorillonite and smectite kaolinite in few depth zone indicates the transformation of clays from their original structure (Millot, 1970). The role of oxidation and reduction phenomena is significant in clay formation and clay diagenesis.

Scanning Electron Microscopy (SEM) and Energy-dispersive X-ray spectroscopy (EDX) analysis

Scanning Electron Microscopy (SEM) analysis has been carried out to support the petrographic and mineralogical results. A total of 50 core samples were analysed using SEM and EDX, the representative images are described here (Plate 3.7 to 3.10). The compaction effect of the shale was envisaged in SEM images where ductile clays are trending parallel. Most of the quartz and muscovite framework grains are embedded in clayey matrix (Plate 3.7). Organic matters are seen as flakes (Plate 3.8) and as pore filling materials (Plate 3.10). Iron nodules are clearly seen in the SEM images (Plate 3.8E). The silica occurs as overgrowth grains or recrystallized silica as major component of the siliceous shale unit (Plate 3.9).

The EDX analysis was used to map the elemental ratio and based on that the probable minerals are identified. The analysis reveals that Barren Measures shale is composed predominantly silica minerals. The elemental ratio indicates the presence of different minerals, viz: FeCO_3 (siderite), SiO_2 (quartz), KAlSi_3O_8 (Orthoclase/microcline) in B#2; depth interval 115-120m; FeCO_3 (siderite), $\text{CaAlSi}_2\text{O}_8$ (Ca plagioclase) in B#2; 105-110m; $(\text{Mg, Fe})_6(\text{Al, Si})_4\text{O}_{10}(\text{OH})$ (Chlorite), $\text{CaAlSi}_2\text{O}_8$ (plagioclase), KAlSi_3O_8 (Orthoclase / microcline) in B#2; 105-110m; and SiO_2 (quartz), KAlSi_3O_8 (Orthoclase / microcline) in plate 3.10E. The results support the XRD data.

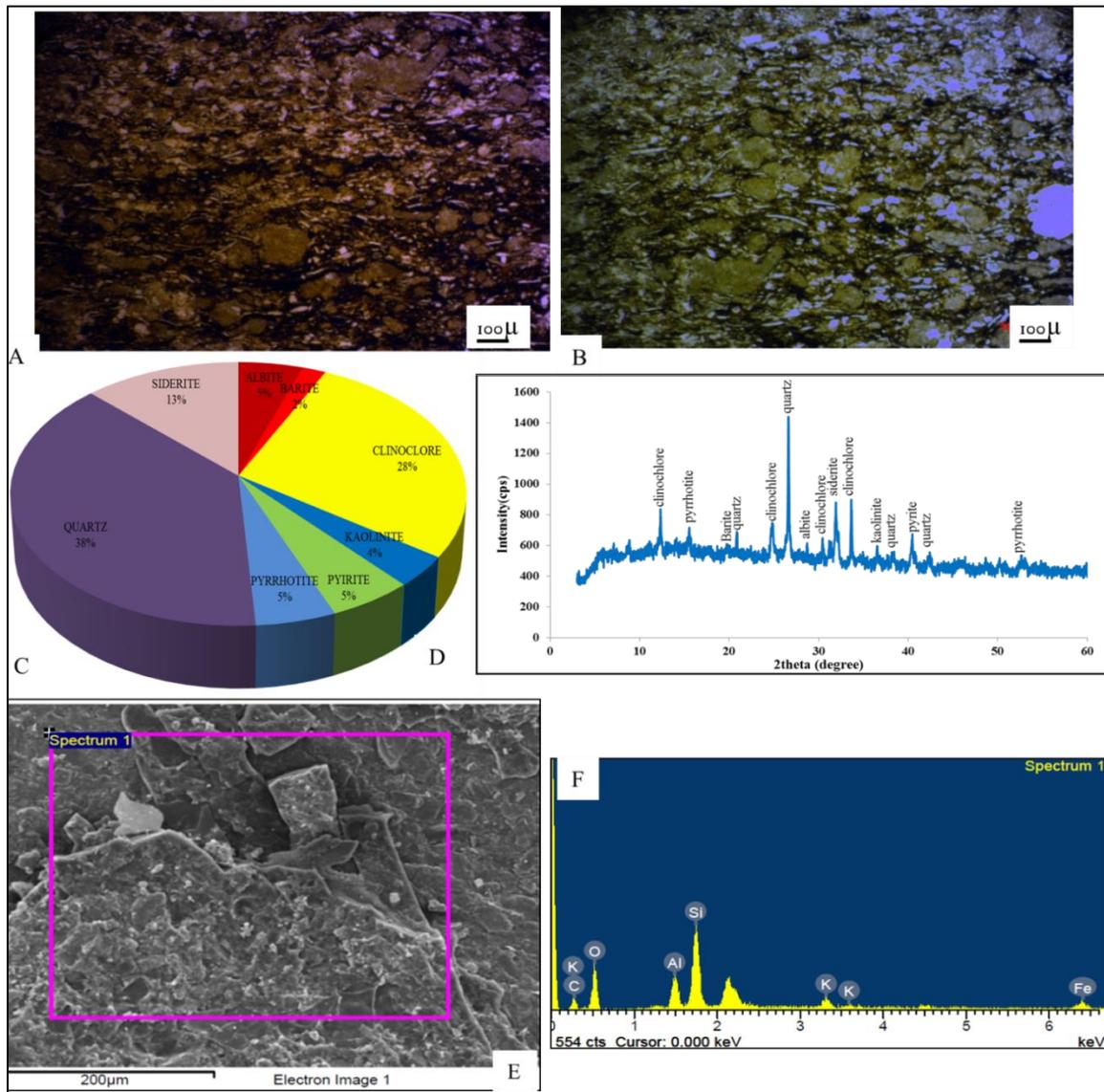


Plate 3.7 Photomicrograph shows the compaction effect on the rock. (Depth interval 115-120m; Borehole: B#2)

- A. The ductile clays are well oriented and trending parallel.
- B. Image A under crossnicol.
- C. Pie diagram of semi quantitative mineral composition,
- D. X-Ray diffractogram showing the mineral peaks,
- E. SEM image indicates fissile laminates shale, matrix is compacted, and grains are coated with clay.
- F. EDX analysis confirms the presence of carbon and iron.

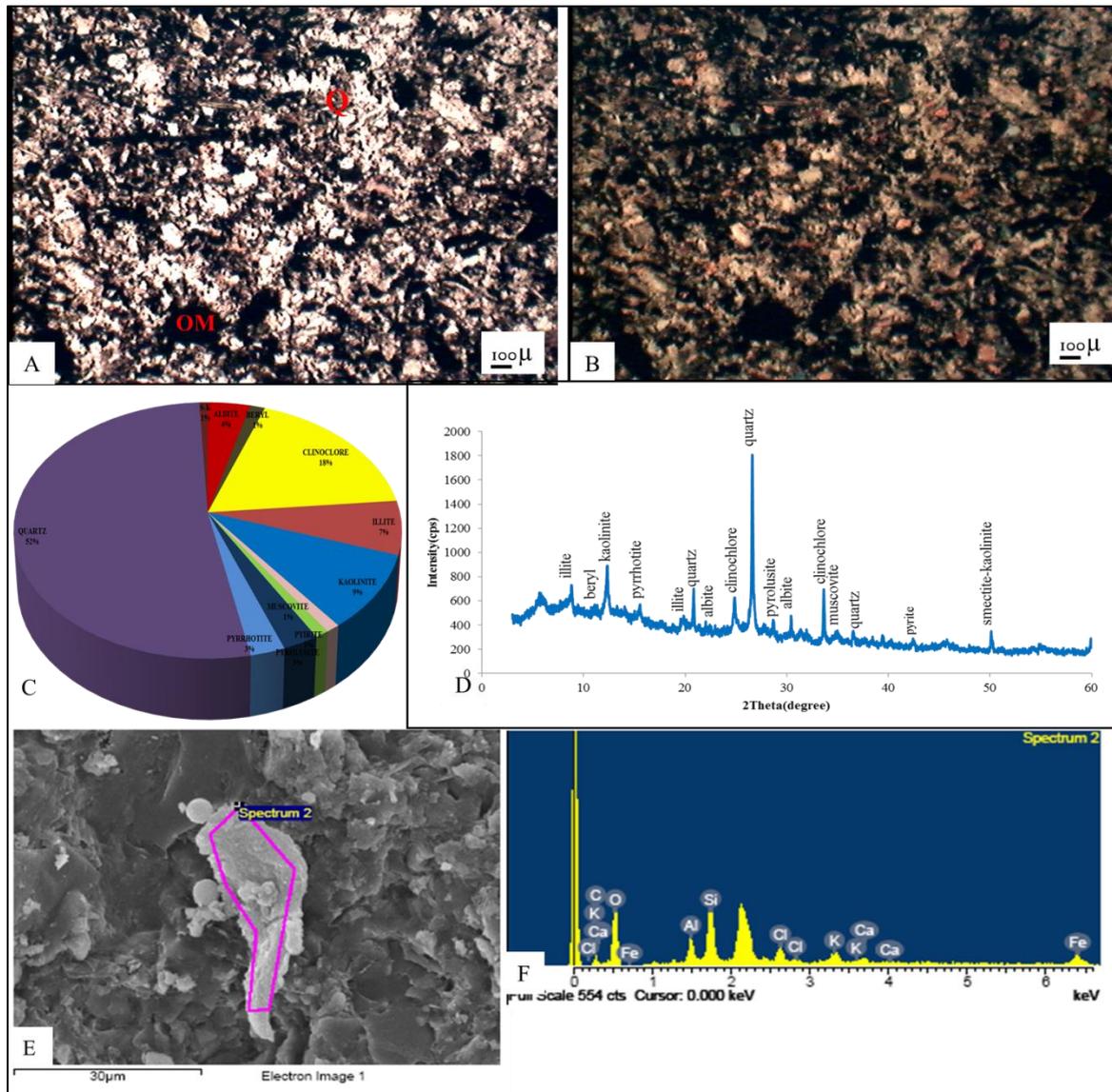


Plate 3.8: Photomicrograph shows ironstone shale; (Borehole: B#2, Depth 105-110m)

- A. Both flaky and rounded organic matters are present.
- B. Image A under crossnicol.
- C. Pie diagram of semi quantitative mineral composition,
- D. X-Ray diffractogram showing the mineral peaks,
- E. SEM image iron ooids formed diagenetically in situ in colloidal clay-iron hydroxide rich sediments by dissolution and precipitation.
- F. EDX analysis confirms the presence of carbon and iron,

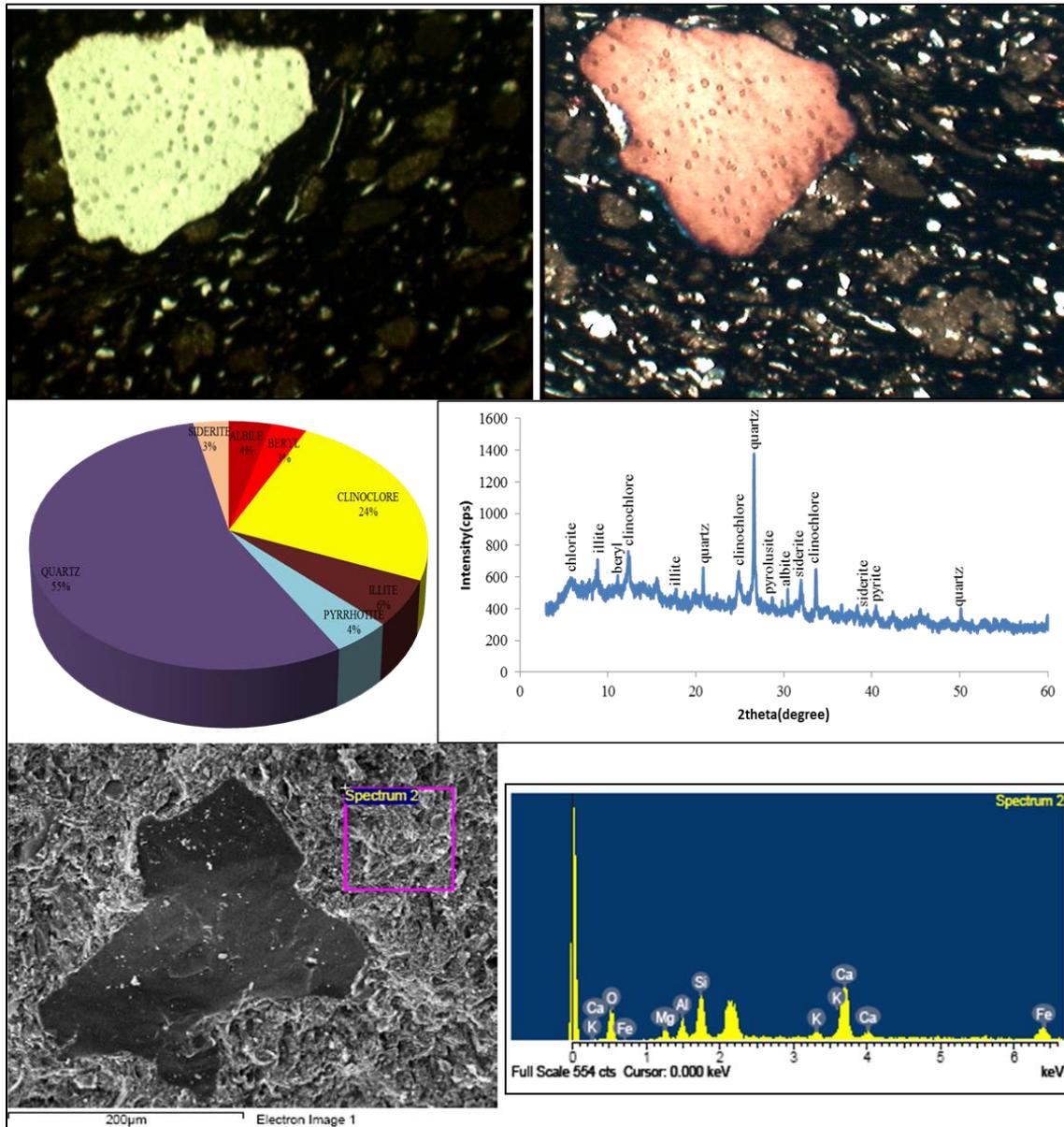


Plate 3.9: Photomicrograph of Barren Measures shale (Borehole: B#4depth 321)

- A. Coarse grains along with well sub rounded fine grains
- B. Image A under crossnicol. Quartz overgrowth is clearly seen, a few micropores are developed on the quartz grain due to dissolution activity
- C. Pie diagram of semi quantitative mineral composition,
- D. X-Ray diffractogram showing the mineral peaks,
- E. SEM image indicates the grains are under compaction.
- F. EDX analysis yields the major elements Si, Ca, Al, and K, with a minor amount of Mg. The Fe detected here is probably due to amorphous iron oxide coatings.

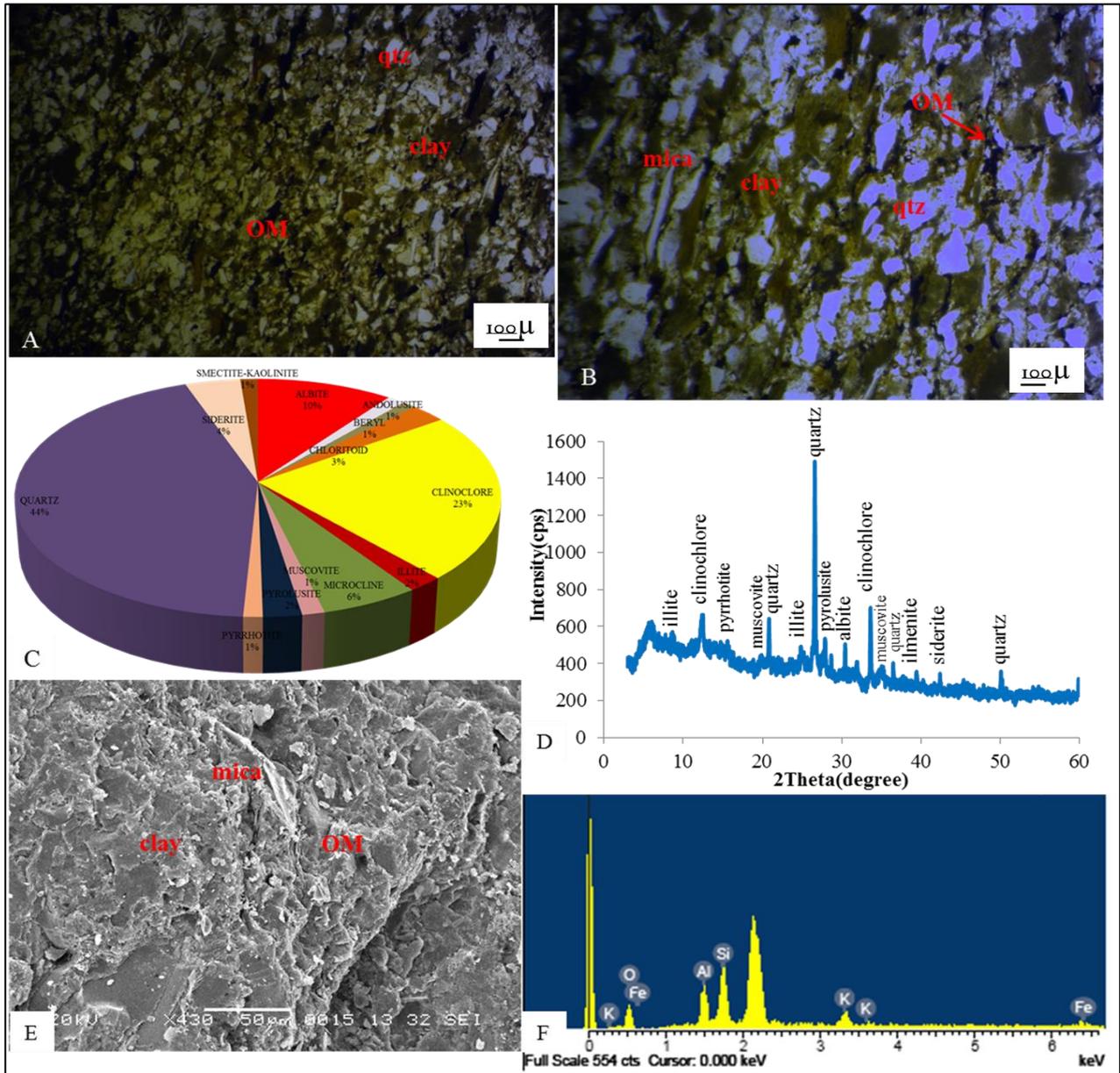


Plate 3.10 Photomicrograph shows the well sub rounded fine grains oriented parallel (B#1, depth 125m).

- A. Pore filling organic matters are present
- B. Image A under crossnicol.
- C. Pie diagram of semi quantitative mineral composition,
- D. X-Ray diffractogram showing the mineral peaks,
- E. Clay rich matrix, micropores are visible.
- F. EDX analysis.

The Barren Measures Formation has been affected by different, early and late, diagenetic processes, Dissolution of different types of cements and unstable detrital grains generate secondary porosity in Barren Measures Formation. The diagenetic effects are clearly observed in different depth levels and few identical SEM images were presented in plate 3.11.

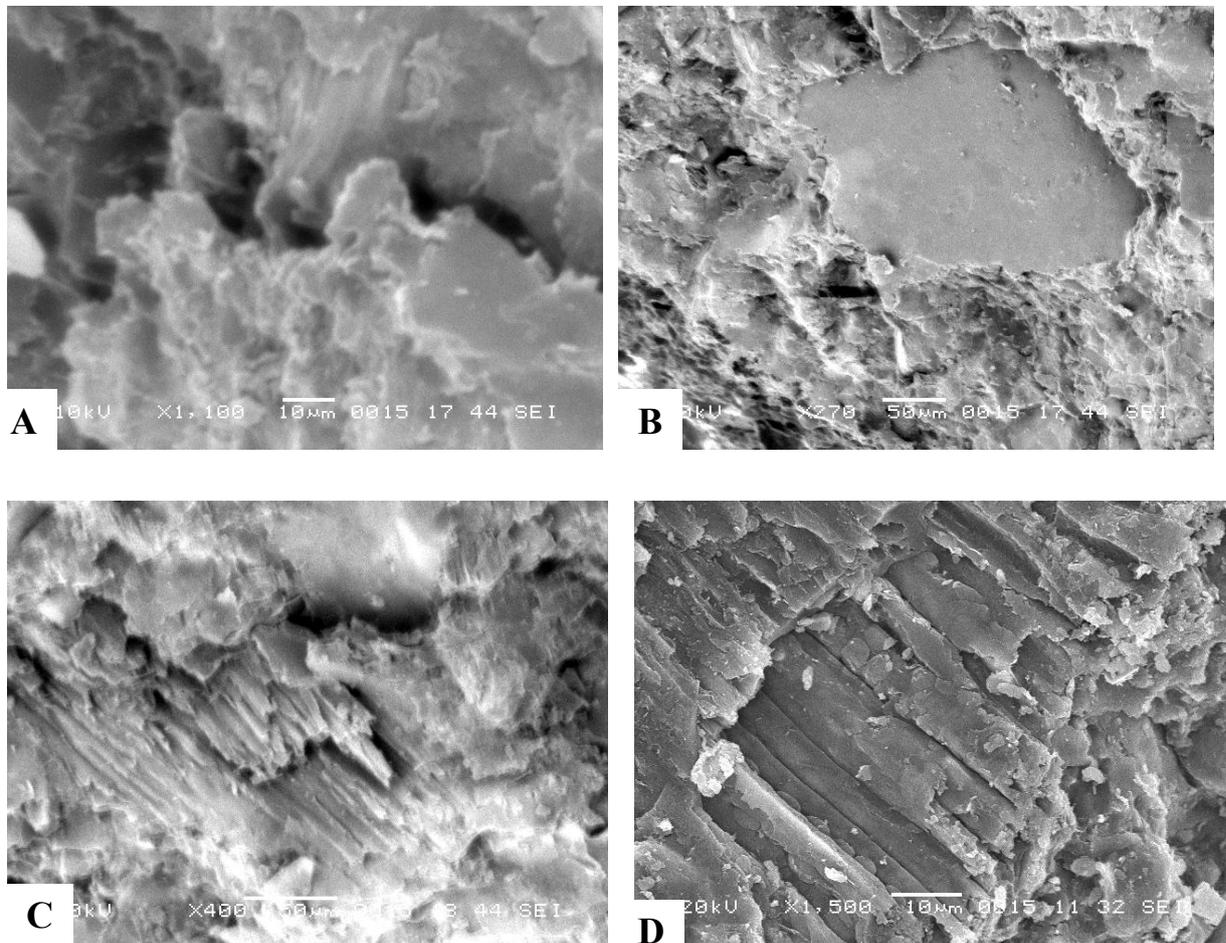


Plate 3.11 SEM images showing diagenesis effects.

- A. *feldspar diagenesis, depth 301-305; Borehole: B#4;*
- B. *Quartz grain alteration under pressure. Depth 301-305; Borehole: B#4;*
- C. *Alteration of feldspar in to clay due to dissolution activities. Depth 310-315m; Borehole: B#4.*
- D. *Feldspar laths*

