Addis Ababa University

College of Natural and computational Sciences

School of Earth Sciences

HYDROCARBON SOURCE ROCK POTENTIAL EVALUATION OF BOKH SHALE OF CALUB AREA IN THE OGADEN BASIN, SOUTHEASTERN ETHIOPIA

A thesis submitted to school of Earth Sciences in partial fulfillment of the requirements for the degree of Master of Science in Petroleum and Coal Geology

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ADDIS ABABA UNIVERSITY
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Declaration

I hereby declare that this thesis is my original work and has not been presented for a degree in any other university, and that all sources of material used for the thesis have been duly acknowledged.

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Acknowledgement

Abstract

The Ogaden Basin is situated in the Southeastern part of Ethiopia. It is the largest sedimentary basin with thick successions and has got an attention for petroleum exploration since the mid of twentieth century. Bokh shale is one of the source rocks in this basin and it is the most targeted formation as it is considered to be a source for Hilala and Calub gas deposit. The previous petroleum exploration and development activities were focused on the regional geology, basin evolution, as well as biostratigraphy of the basin but a little is done about the hydrocarbon potential. Therefore, this study iss proposed to determine the hydrocarbon potential of the source rock and to establish the depositional environment and to infer the provenance of Bokh shale. In this study a total of 12 core samples of Bokh shale from Calub 2, 3 and 5 were selected. The organic geochemical of six samples were analyzed using Rock-Eval pyrolysis analytical method. The results were used to determine organic richness and hydrocarbon generation potential, and kerogen type. The organic petrography of the two samples were studied, the results then applied to establish thermal maturity of the Bokh shale. The major, minor and trace elements of four samples were investigated using X-ray fluorescence (XRF) techniques. The results were used to infer the depositional environment and provenance of the source rock. From the analyzed samples the average total organic content (TOC) of the Bokh shale obtained is 0.521 wt% and ranges from ~0.5 to 0.56 wt%. The pyrolysis result indicated S1 ranging from 0.02 to 0.23 mgHC/g rock with an average of 0.076 mgHC/g rock and that of S2 is from 0.05 to 0.27 mgHC/g rock with an average of 0.116 mgHC/g rock. The average generation potential of the source rock is 0.192 mgHC/g rock. The vitrinite reflectance of Calub 3 exhibits 1.79 Ro (random reflectance) whereas Calub 5 indicates 1.3 Ro. The overall organic richness and kerogen type of Bokh shale signify fair carbon quantity and poor gas generation potential. The studied sample shows kerogen type IV, i.e. it is dominated by inertinite macerals that generate little or no hydrocarbons upon further maturation. The examined vitrinite reflectance of samples suggest late maturate (Calub 5) to over-mature (Calub 3) organic matter in the Bokh shale. The V to Ni ratio of Calub 3 indicates it was formed of mixed marine and terrestrial organic matter while the Calub 5 was formed of marine organic matter. The examination of ratio of V to V+Ni suggests both samples were deposited under sub-oxic environmental condition. The Zr/Cr, La/Sc, and La/Co ratio indicates both samples were sourced from felsic preexisting rock.

Keywords: Ogaden Basin, Bokh shale, Calub, Quality and Quantity, Thermal maturity

List of Acronyms

Abbreviated terms of Pyrolysis

TOC   Total organic carbon

GP   Generation Potential

OI   Oxygen Index

Tmax   Maximum Temperature

 HI   Hydrogen Index

PI   Production Index

Other abbreviated terms

SPEE   Soviet Petroleum Exploration Expedition

MoME   Ministry of Mines Ethiopia

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CHAPTER 1

1   INTRODUCTION

1.1   Study background

The sedimentary basins of Ethiopia cover a significant portion of the country and there are five distinct sedimentary basins; namely: Ogaden (350,000 km2), Blue Nile (63,000 km2), Gambela (17,500 km2), Mekele (8000 km2) and Southern Rift Basins (N-S striking) (Assefa, 1988; Barnes, 1976; Beauchamp et.al., 1990 and Worku and Astin 1992). The Ogaden, Abay and Mekele basins are presumed to be intracontinental rift basins formed as a result of extensional stresses induced by the break-up of Gondwanaland in Upper Paleozoic.

The Ogaden Basin is located in the southeastern part of Ethiopia (Fig.1.1), and it occupies an area of about 350,000 sq. Km. The basin's formation is related with the Permo-Triassic break-up of the mega continent —Gondwanaland. This Paleozoic-Mesozoic basin covers large area in the southeast Ethiopia and the basin geometry is characterized by deep, asymmetrical grabens separated by internal highs (MoM, 2011). The intra-cratonic Ogaden basin was developed in response to rifting of the NE-SW to ENE-WSW trending Calub Saddle in the east of the basin, NNE-SSW oriented Mandera-Bodle Rift known as the Bodle Deep in the southern part, and NW-SE trending Blue Nile rift in the NW of the basin. This tri-radial rifting was active during Late Palaeozoic to Mesozoic times (Hunegnaw *et al.*, 1998). The Paleozoic and Tertiary sedimentary successions reach up to 10,000 m. The oldest sediments in the basin are Karoo sediments (Permian to Lower Jurassic). This continental sediment deposit has an average thickness of up to 1km. The sequence is divided into four formations, from oldest to youngest are Calub sandstone, Bokh shale, Gumburo sandstone, and Adigrat sandstone. Thick Permian to Cretaceous sequences has proved petroleum potential, which primarily occurs in the South West and Central parts of the basin**.**

The Ogaden Basin has long been considered prospective for petroleum and therefore it has attracted the attention of several oil companies involving in the exploration work. In Ethiopia, petroleum exploration was started in 1920 by Anglo American, the British arm of the Standard Oil Company of New Jersey (John, 2016). Then, in 1945 Sinclair Petroleum Co. carried out aerial photography, surface geological surveying, and mapping, and gravity, magnetic and reflection seismic surveys, but reported oil show in 1952 (Hunegnaw, 1998). The exploration companies, Sinclair and Elwerath of Germany Oil Company later in 1959, have confirmed the existence of oil source rock in the Ogaden basin by drilling and seismic

surveying. Since then several petroleum exploration companies actively participated in search of hydrocarbon deposits in the Ogaden Basin. Forty-six exploration wells drilled so far confirms the Ogaden basin as a petroliferous basin having all the essential elements of a petroleum system. And in the few wells, oil and gas shows were reported (Hunegnaw, 1998). As in the Assefa (1988) the Permian Bokh Shale, Hamanlei Limestone, and Urandab Shale are potential organic-rich source rocks identified so far in the Ogaden Basin, whereas Permian Calub sandstone, Adigrat sandstone, and Hamanlei carbonates are the potential reservoirs of the Basin (John, 2016).

According to Wolela (2008) and Worku and Astin (1992) the Bokh shale, which is estimated to be 450m thick, is characterized by predominantly dark grey and minor dark green to reddish brown shales with interbeds of siltstone and fine sandstone and is thinly laminated. The formation grades upward from the clay-size material into bioturbated siltstone and fine sand. The dark shale has variable content of fauna and flora. It is assumed as a source for Hilala and Calub gas deposit. The Permian to Early Triassic Bokh shale revealed petroleum potential up to 7 kg HC/t. This study will evaluate the hydrocarbon potential of Bokh shale by using the most recent geochemical methodologies to determine its generative potential.
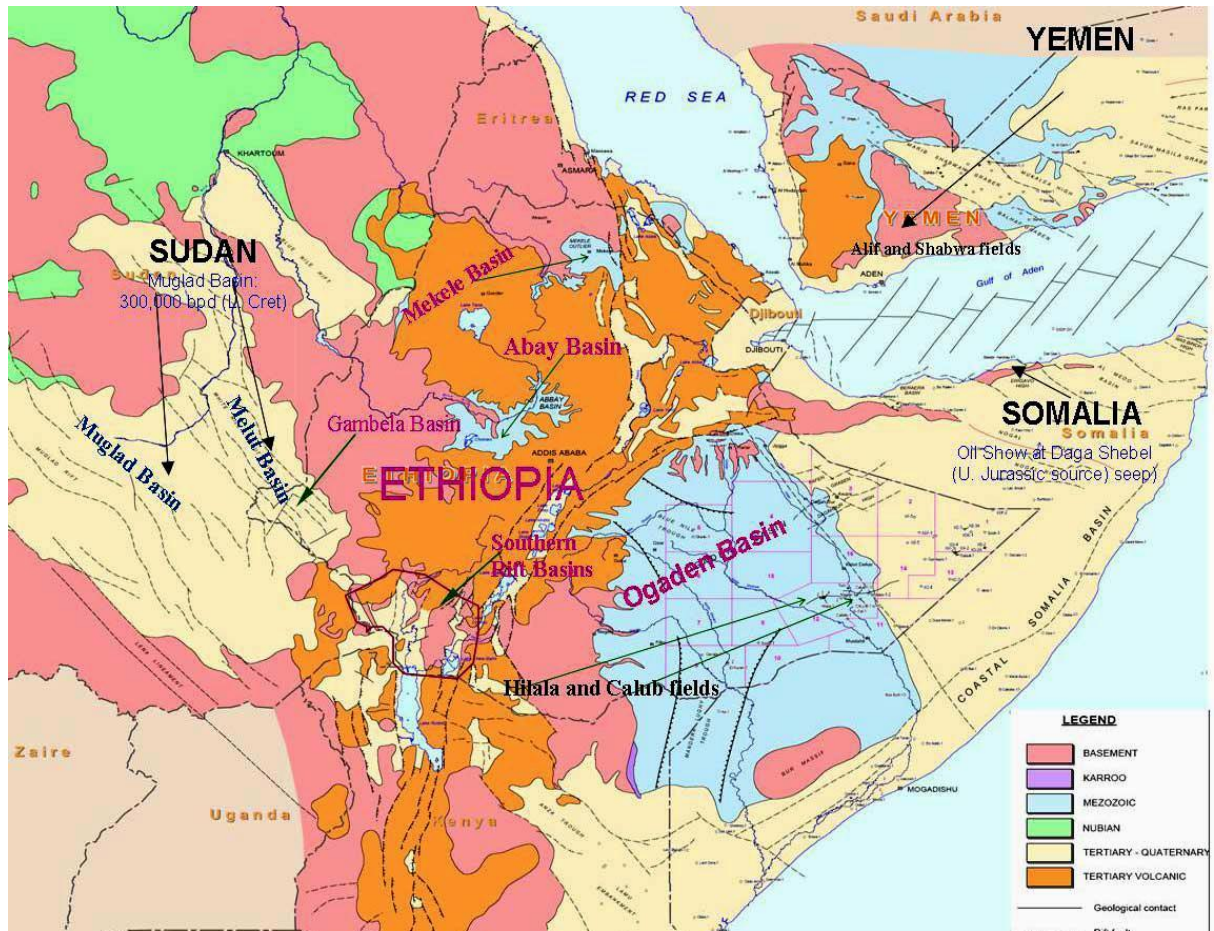
Figure 1.1 Prospective Sedimentary Basins of Ethiopia (MoME, 2011)

1.2   Study area

1.2.1   Geographic location

The study area is situated in the eastern part of Ogaden basin and approximately 970 km far from Addis Ababa along the major road. It is bounded between $06^008'00''$ and $06^011'00''$N and $44^029'00''$ and $44^033'00''$E and located within the Korahe zone which is bounded from North by Deghabor zone, from east by Warder Zone and from west and south by Gode zone. This road takes either from Addis Ababa-Harar-Jijiga-Deghabor-Korahe or from Addis Ababa-Harar-Jijiga-Warder-Korahe. Most of the region is plain with elevation ranging from 300 m to 600 m, with a steady rise to north, west and southwest to more than 1km above sea level (Assefa, 1998).
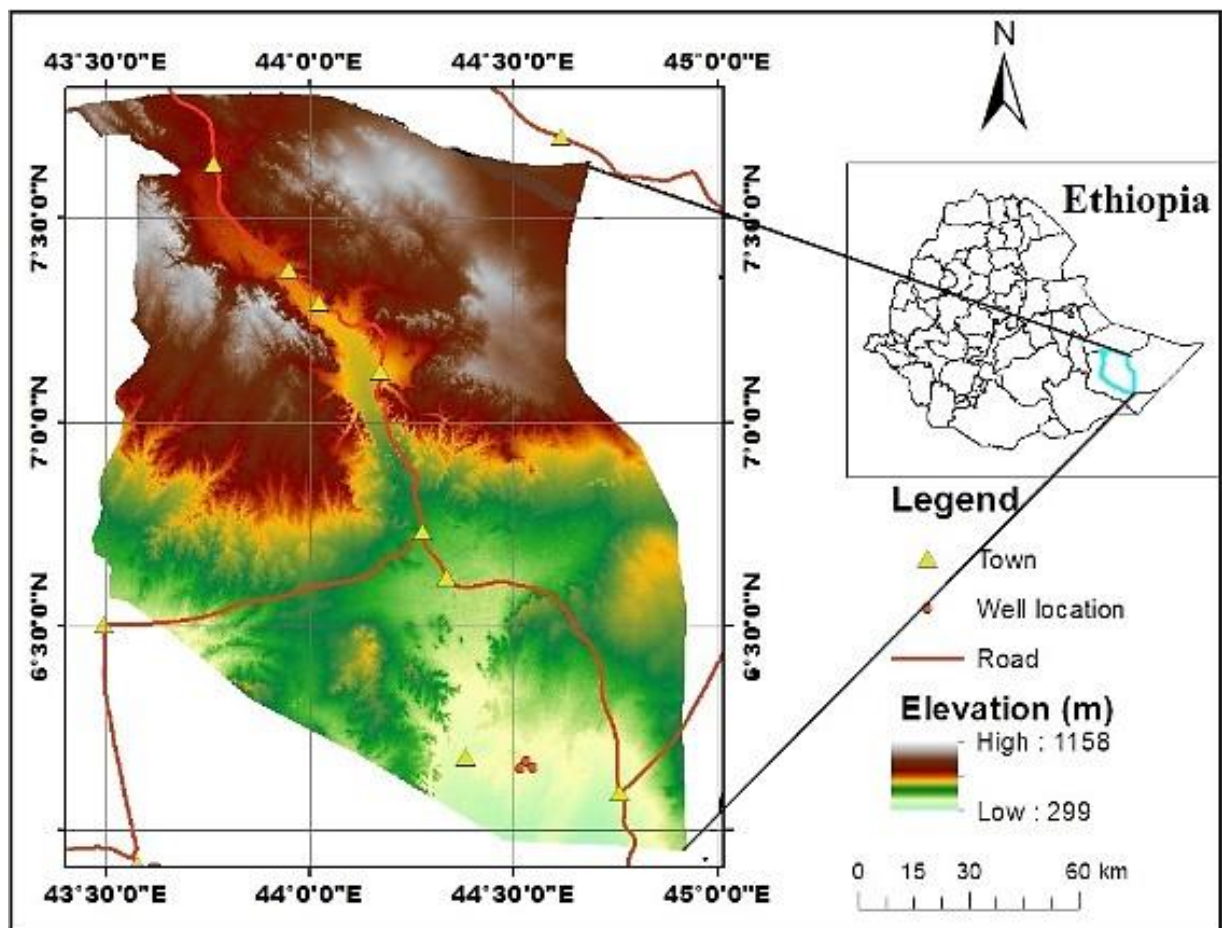
Figure 1.2 Location map of study area

1.2.2 Climate

The climatic zones in the Ogaden and adjacent region are described (Ethiopian Mapping Authority, 1988; Lemma, 1996). The bereha zone, hot arid, covers the region below 500 m elevation, where average annual rainfall is less than 400 mm, resulting in sparse vegetation with the extensive bare ground. The kolla zone, warm to hot semiarid, covers the region between 500 and 1,500 m in elevation, where the average annual rainfall is 600–800 mm. The higher regions of the Somali Plateau are classified as weyna dega (1,500– 2,500 m.a.s.l.; warm to cool semi humid) and dega (>2,500 m.a.s.l.; cool to cool humid). Under the Köppen classification, the Ogaden is classified as hot arid (Bwh) and hot semiarid (Bsh). In the high country above 2,000 m (beyond the Ogaden sensu stricto), the climate is warm temperate (Cwb). For all intents and purposes, Koppen zones Bwh and Bsh correspond to the bereha and kolla zones.

Figure 1.3 climate zones, southeast Ethiopia (after Lemma, 1996 and EMA, 1988)

1.2.3   Physiography of the area

There seems three informal physiographic features division in the province. These are Genale and Shebelle drainage basins and the Eastern Slope and Plains. The drainage basins include outstanding upstream canyons that evidenced the vertical movements that have accompanied the succession of rifting events in the Ethiopian Rift, Afar, and the Gulf of Aden. In contrast, the Eastern Slopes and Plains is gentle lying over hundreds of kilometers to the southeast and are covered by sheets of red sands.
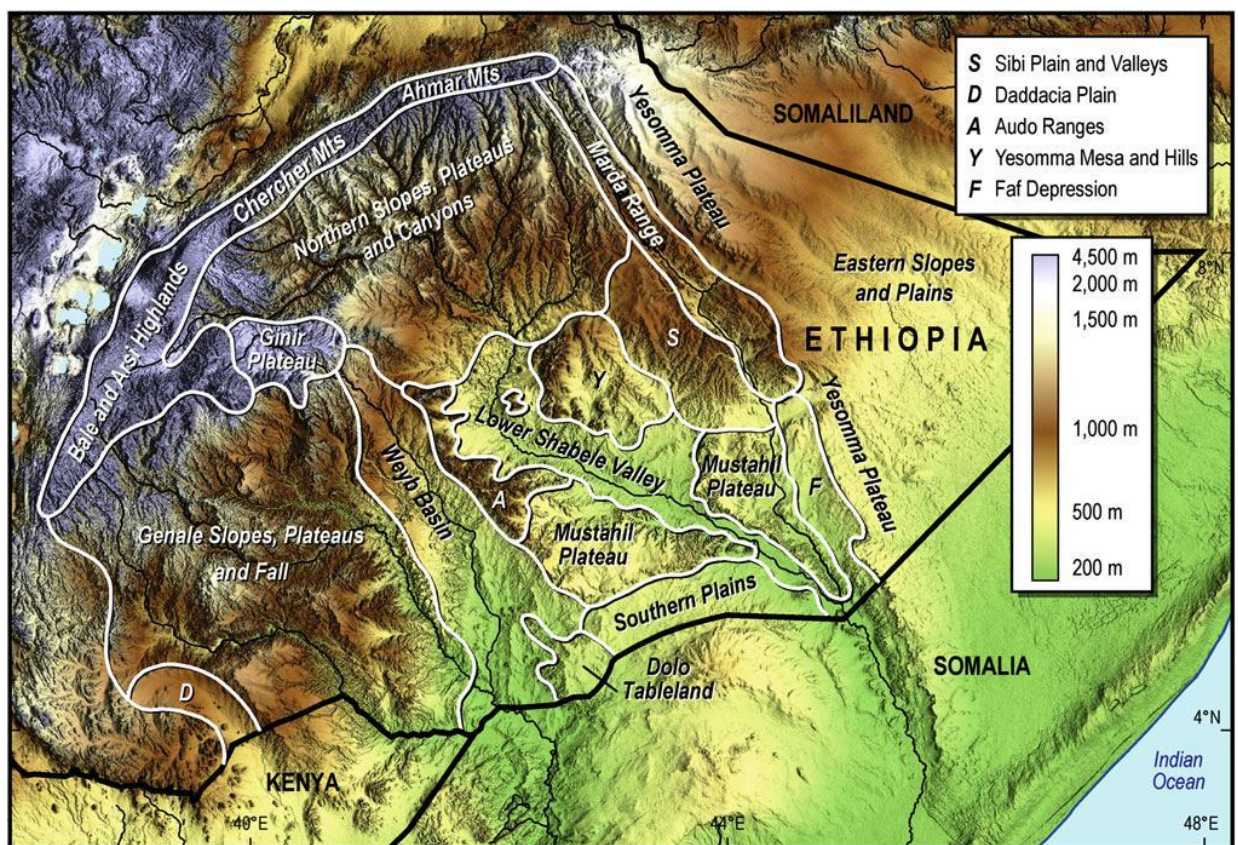
Figure 1.4 Digital elevation model, southeast Ethiopia, showing informal physiographic subdivisions (after Mege et al., 2015)

1.3 Previous works and exploration activities

Beginning from early 20th century different studies have been carried out in the Ogaden basin from different perspectives. According to Purcell, 1976 the first oil seep in Ethiopia was reported in 1860. Early reports (Molly, 1928; Belluigi, 1937) also refer to seeps in the Ogaden Basin: in the Gara Mulatta Mountains near Harar, in the Fafan and Gerer river valleys, and near Jijiga. Hunegnaw et al. 1998 have summarized the previous exploration activities by Petroleum companies as following:

In 1945 Sinclair Petroleum Co. carried out aerial photography, surface geological surveying and mapping, and gravity, magnetic and reflection seismic surveys. Later in 1949 and 1952 the company drilled Gumburo-1(3087m) and Galadi-1(2769m) wells respectively but encountered oil show in Galadi-1 only. Elwerath, a German oil company carried out an aerial photographic survey with further geological mapping in 1959; shot 1925 line km of seismic section profile between 1961 and 1963 and also carried out Gravity and Magnetic surveys. Elwerath drilled two wells between 1963 (Abred-1) and 1965 (Bokh-1) but no significant oil and gas show.

In 1969 Tenneco Oil Co. carried out aerial photographic studies and geological surveying and drilled Elkuran-1 (3189m), Elkuran-2 (2015m) and Calub-1 (3685m) exploratory wells in 1972 and 1973. Oil and gas reported in both Elkuran wells and gas discovery in Calub-1. Between 1973 and 1974 the company drilled five further wells Magan-1 (3575m), Callafo-1 (3242m), Bodle-1 (3911m), Hilala-1 (4116m), and Gherbi-1 (1976m) but oil and gas show encountered only in Hilala-1.

In 1973, the Whitestone Petroleum Co. and the Voyager Group (Voyager Petroleum Ltd., Polar Bear International Petroleum Ltd., Houston Oil Ltd. and the Cardinal Petroleum Co.) conducted aerial-photographic, geological and gravity surveys over 77000 sq.km and 54000 sq.km areas respectively. In addition, Whitestone carried out an aeromagnetic survey covering 16,000 sq. km in 1976.

The Soviet Petroleum Exploration Expedition (SPEE) was established in 1980 to explore the area between Shillabo and Hilala in the central Ogaden Basin. SPEE shot 1,544line-km of seismic profiles and drilled four wells: Shillabo-1(2,900m), Hilala-2 (2,400m), Hilala-3 (1,760m) and South Calub-1(1,700m). Oil and gas shows were reported in the first three of these wells. An intensive seismic survey (2,777 line-km) was carried out by SPEE between 1984 and 1987, and during the same period, five further wells were drilled: Faf 1 (3,446m),

Magan-2 (4,306m), Calub-2(3,732m), Calub-3(3,690m) and Tulli-1 (4,010m). The earlier Culub-1 gas discovery was confirmed by Calub-2. After renewal of the agreement, the SPEE continued its operations and drilled seven appraisal wells on the Calub field (Culub-4 to Culub-10).

In 1990, Maxus Energy and Hunt Oil Co. were awarded exploration acreage in the NE and SW of the Ogaden Basin, respectively. In 1995, Hunt Oil drilled a dry well (Genale-1) near the Genale river oil seep in the west of the basin, and Maxus relinquished its concession after conducting seismic work (1,160line-km).

Other than exploration by Petroleum Company different works have been done by many authors. Among these workers, Assefa (1988) by using Lopatin method calculated time-temperature indices of rock maturation of Ogaden Basin and suggested that during the Phanerozoic eon, i.e. Paleozoic and Mesozoic rocks, the environment were favorable for petroleum (gas) generation. Furthermore, Assefa (1988) indicated that Bokh shale and Hamanlei formation are potential source rocks, and predicted the time they started to generate oil. Accordingly, Bokh shale begun to generate oil around 220 my and Hamanlei formation commenced some 170 my ago. In the study of Karoo sediments of Ogaden Basin, Worku and Astin (1992) stated the dark shale has variable organic carbon contents up to 5% and thermal organic maturity varies widely with the Bokh area. Following the extensive literature, oil company reports, research review, and core sample analyses, Shigut (1997) concluded Urandab Shale Formation, Upper Hamanlei carbonates, Transition Zone formation are the good potential source rock and Bokh shale is a good gas source. The Stratigraphic and structural frameworks related to hydrocarbon potential of Ogaden basin were reported in detailed by Hunegnaw *et al.* (1998). From this point, it has been concluded that the basin has good quality source rocks bearing fair to good hydrocarbon potential and complete petroleum system and processes. Through core studies, geochemical and log analysis (Beicip-Franlab, 1985, 1998; Robertson Research, 1986) has identified petroleum systems. Source rocks are organic rich Bokh shale, Transitional zone, and Urandab formation; Reservoir rocks: sandstone in Calub and Adigrat formation and carbonates in the Hamanlei formation; Traps: both structural and stratigraphic traps. The overall organic richness of the source rock was done by Wolela (2008) is that the TOC values of Bokh shale range from 0.5 to 1.5 %. The cross plot of hydrogen index versus Tmax values indicated the Bokh shale, Transitional facies, and Urandab Shale are excellent types of source rocks.

Even though different oil and gas shows were reported in various wells, the hydrocarbon deposits discovered yet are only in the Hilala and Calub field. The reserves of 2.7 TCF condensates and 1.3 TCF gas deposit are estimated in the Calub and Hilala field respectively (Wolela, 2008 and Hunegnaw et al., 1998). This the total reserve of 4Tcf (reserve at Calub field is 2.7 Tcf and at Hilala field 1.3 Tcf) are recently awarded to a company named PetroTrans (Hong Kong-based Company) along with other blocks in the Ogaden Basin.

1.4 Hydrocarbon source rock evaluation

1.4.1 Quantity of organic matter

The quantity and quality of organic matter preserved during diagenesis of sediment determines the generative potential of the source rock. Quantity is determined by amount of organic input, the degree to which it is preserved either as primary or secondary biogenic matter, and by its dilution with inorganic mineral matter (Walters, 2007). Petroleum is a generative product of organic matter disseminated in the source rock, and the quantity of the petroleum should be corelatable with the organic richness of the potential source rock (Tissot & Welte, 1984).

The organic richness and potential of a rock sample is evaluated by measuring the amount of total organic carbon (TOC) in the whole rock and pyrolysis derived S2 of the rock samples (Peters and Cassa, 1994; Peters, 1986). Rocks which contain TOC less than 0.5 wt% and S2 less than 2.5 mg/g are considered poor source rocks. Samples containing from 0.5 to 1 wt% TOC and S2 from 2.5 to 5 mg/g are fair source rocks, and those constituting TOC from 1 to 2 wt% and S2 from 5 to 10 mg/g are good source rocks. More than 2 wt% TOC and S2>10mg/g are considered very good source rocks (Peters, 1986).

1.4.2 Quality of organic matter

Organic matter deposited in sediments is an extremely complex mixture of many types of organic compounds. Different types of organic matter have different hydrocarbon potentials due to the fact that variation in the chemical structure of organic matter. The organic matter type in a sedimentary rock, among other conditions influences to a large extent the type and quality of hydrocarbon generated due to different organic matter type convertibility (Tissot and Welte, 1984). According to Peters and Cassa (1994) and Jacobson (1991), there are four types of kerogen in sedimentary rocks: Type-I, composed of oil-prone hydrogen-rich organic matter generally in lacustrine and some marine sediment. Type-II also composed of oil-prone hydrogen-rich organic matter mainly in marine sediment. Even though oil is the main product

of type-II kerogen, it actually produces more gas than type-III kerogen (Hunt, 1996). Type-III composed of terrestrial organic matter derived mainly from woody plant material that is low in hydrogen content and generates mainly gas, and Type-IV composed of dead or inert carbon that has little or no generating capacity.

1.4.3 Maturity of organic matter

Maturity is the degree of thermal evolution of the sedimentary organic matter (source rock). Thermal evolution of the source rock changes many physical or chemical properties of the organic matter (Tissot and Welte, 1984). Combining and finding relations between the essential Rock-Eval parameter, Tmax, and calculated Rock-Eval parameter, PI, is a valuable method for indicating the thermal maturity of organic matter (Peters, 1986; Peters and Cassa, 1994; Bacon *et al.*, 2000). Similarly, the cross plot of HI against Tmax indicates thermal maturity of organic matter (Van Krevelen, 1961).

One of the most extensively used parameters for evaluating the thermal maturity of organic matter is vitrinite reflectance (VR), which relies on the property of vitrinite to undergo increasing reflectance with increasing temperature. It is measured under a reflected light microscope (Allen and Allen, 1990). Vitrinite Reflectance is considered a key indicator primarily due to the natural properties of vitrinite, which include consistent physical and chemical changes under increasing thermal stress, and uniform appearance under reflected light microscopy regardless of orientation (Mukhopadhyay and Dow, 1994). Because the effects of increasing thermal stress on vitrinite are progressive and irreversible, vitrinite reflectance can provide important information on the thermal maturation and hence burial history of a basin (Kalkreuth and McMechan, 1988).

1.5 Problem statement

Petroleum was used for many centuries in the ancient civilization of Mesopotamia, Egypt, Persia, China and elsewhere in the world for heating, lighting, road making, and building purposes. Since the ancient history of human being, the exploration and production of petroleum are significantly increased due to high demand for it. Similarly, exploration for petroleum and natural gas in Ethiopia is increasing from time to time. Initial exploration for hydrocarbons in the Ethiopian Ogaden Basin was begun around 1920 by Anglo American, the British arm of the Standard Oil Company of New Jersey (John, 2016). Since then different petroleum exploration companies have been awarded and took part in search of petroleum in the Ogaden Basin which is partitioned into different blocks. Aerial photographs,

surface geological mapping and surveying, magnetic, gravity and, reflection seismic survey method have been conducted in 1945 by Sinclair Petroleum Co. Then in 1952 an oil show encountered in Galadi-1 well. Tenneco Oil Co. drilled three exploratory wells in 1972 and 1973 and reported oil and gas shows in El kuran-1 and-2. Again in between 1973 and 1974 the company reported significant oil and gas shows in Hilala-1. In 1980 SPEE drilled four wells and reported oil and gas shows in Shillabo-1, Hilala-2 and Hilala-3. After drilling additional l5 further wells between 1984 and 1987 SPEE confirmed the earlier gas discovery in Calub-1 by Calub-2 (Hunegnaw et al., 1998). Currently petroleum exploration and development activity indicates a number of international and few local companies are currently undertaking petroleum exploration in various parts of the country. Calub and Hilala gas-condensate fields with estimated total reserve of 4Tcf (reserve at Calub field is 2.7 Tcf and at Hilala field 1.3 Tcf) are recently awarded to a company named PetroTrans (Hong Kong-based Company) along with other blocks in the Ogaden Basin. The occurrence of source rock and petroleum system was verified in the intracratonic Ogaden Basin, however, little was done on the potential evaluation of source rock regional wise by Hunegnaw *et al.* (1998), Wolela (2008), and Shigut, (1997). Therefore, this study is designed to evaluate the hydrocarbon potential of Bokh shale using organic geochemistry and reflectance methods. In addition the study identifies the provenance of the source rock and establishes depositional environment from inorganic geochemistry.

1.6   Objectives of the study

1.6.1   General objective

The study is generally intended to evaluate the hydrocarbon potential of Bokh shale of Calub area in Ogaden basin through geochemical analysis. The purpose of the   geochemical analysis is that it is used to measure a number of organic components in the source rock.

1.6.2   Specific objective

The specific objectives of the study are:

- Measuring the TOC (quantity, or amount of organic matter) amount
- Characterizing the kerogen types (quality of organic matter), and
- Identifying the maturity level of source rock.

1.7   Significance

Large sedimentary basin in East Africa is located in Ethiopia like Ogaden basin is one to mention. This basin has similar geology with Middle East country where petroleum is being produced. Many kinds of literature were written about Ogaden basin by scholars and petroleum operators. Various exploratory wells drilled so far to understand the resources the basin constitutes; seismic surveys were also conducted to identify the structure and stratigraphy of the basin. After many years of exploration, the basin was found to be the area of promising hydrocarbon potential and its reserve was calculated. This study has dealt with geochemistry of Bokh shale and hence evaluated the amount of organic constituents source rock has, type of organic matter shale is made of and, thermal maturity level at which organic matter was transformed to the organic compound. The study has also assigned the possible category of generative potential of the source rock.

CHAPTER 2

2   REGIONAL GEOLOGICAL SETTING

2.1   Introduction

The intracratonic Ogaden Basin (Late Palaeozoic-Mesozoic), which covers approximately one-third of Ethiopia, is bounded to the North and NW by the Ethiopian portion of the Miocene-Quaternary East African Rift, and to the west and SW by basement outcrops whereas to the South, East and NE, the Ethiopian Ogaden Basin adjoins a sedimentary basin in Somalia (Hunegnaw *et al*., 1998).

2.2   History of Ogaden Basin and structural development

2.2.1   Tectonic evolution of Ogaden Basin

The East African region has been affected by two major phases of rifting (Worku and Astin, 1992). The first phase related to Late Paleozoic to Jurassic and the second rifting relates to the formation of the East African rift system (Cenozoic to Recent).

The first phase was the widespread rifting in Karoo times (Late Carboniferous to Early Jurassic) which stretches from Ethiopia to South Africa and corresponds to the initiation of the Gondwanaland break-up (Norton and Sclater, 1979; Bosellini, 1989) in that further subsidence took place. This subsidence combined with sea-level fluctuations and produced cyclic patterns of shallow- marine carbonates, shales, evaporites and minor clastic deposits.

The development of the Ogaden Basin is related to the break-up of Gondwanaland (Hunegnaw *et al*., 1998). Extensional rifting within Gondwana occurred during the Late Paleozoic (Late Carboniferous to Permian), through the Triassic, and into the Jurassic (John, 2016). Pre-breakup extension was occurring and marine invasion was beginning in the resultant developing lows, while rift basins were beginning to form inland. In northeast Africa, including Ethiopia, fluvial-lacustrine sediments sourced from exposed highlands began to fill the rift basins (John, 2016). According to Hunegnaw et al. (1998) in the Ogaden basin the sediments deposited are associated with different phases of deformation, i.e. the pre-rift sediments (Calub formation), initial rift sediment (Bokh and Gumburo Formation), early rift sediments (Adigrat sandstone Formation and Lower Hamanlei Formation), Syn-rift sediments (Middle Hamanlei) and post-rift sediments (Antalo Limestone and Ambaradam Formation).

At the same time as the break-up transgression in the Middle-Late Jurassic, a passive margin began to develop in the Ogaden area. This resulted in alternating regressions and transgressions, which continue at present in response to down/upwarping of the Ethiopian-Somalian platform. During this period, pre-existing faults were reactivated (Hunegnaw *et al*., 1998).

The opening of the South Atlantic Ocean was initiated during the Cretaceous, and a number of shear zones were developed along and within the East African continental margin (Bosworth, 1994). During this post-rift period, regressions and transgressions alternated over the Ogaden platform and the corresponding sediments deposited.

2.2.2   Structure of Ogaden Basin

Ogaden basin has several structural highs and lows separated by normal faults (kent, 1974). Cannon *et al*. (1981) stated that continental Karoo deposition was initiated and controlled by major faulting of the Paleozoic/Mesozoic triple-junction rift system developed in East Africa. Purcell (1976), BEICIP (1985), Hunegnaw *et al*. (1998) and John (2016) concluded that the Ogaden Basin developed as part of a triple-junction rift system during Late Paleozoic-Mesozoic are the Mandera- Lugh Deep, the Calub saddle of the Ogaden Basin, and the Blue Nile rift to the northwest (Fig. 2.1). Thick Karoo sediments filled the rift troughs. These rifts initiated the Mandera-Lugh Basin of southeastern Ethiopia and the Karoo rift hosting the Calub Gas discovery within the Ogaden Basin of Ethiopia.

a.   The NE-SW to ENE-WSW trending Calub Saddle in the east of the basin includes the North Shillabo half-graben. These structures formed as a result of NW-SE oriented extension. The Calub Saddle is separated from the coastal Somalia Basin by the Bur High.
The North Shillabo half-graben progressively deepens from north to south, with a maximum sedimentary thickness of 7,500m next to the major ENE-WSW trending Shillabo fault which separates the half-graben from the Calub Saddle. The Calub Saddle comprises a "deep" zone adjacent to the Bodle Deep, where the basement is about 5,500-m deep; a steeply-dipping flexure zone; and a relatively high area to the south, where basement depths range from 4,200 to 2,500 m in the direction of the Bur High.

b.   The SSW-NNE oriented Mandera-Bodle Rift (known as the Bodle Deep in the southern part of the basin), which is bounded by transcurrent faults.

A thickness of over 10,000mof sedimentary rocks is present in the deepest (southernmost) part of the Bodle Deep, which has a steeply-dipping western flank deformed by north-south and NNE-SSW trending faults and a gentler eastern flank. The floor of the basin rises progressively northwards; the depth to basement is around 6,100m at the Bodle-1 well (Fig. 2.2), and 5,000m at the junction of the tri-radial rift system.

c.   The SE portion of the NW-SE trending Blue Nile rift in the NW of the basin.

At the southern end of the Blue Nile Rift, depth- to- basement progressively increases SE wards, from around 900m at the Gherbi-1 well location to about 5,000m.

Like other similar basins in NE Africa, Ogaden Basin is dominated by non-compressional tectonics. The two major fractures dominating in the Ogaden Basin are: Marda Fracture Zone and the Kuran Fracture Zone dominate the structural framework (Assefa, 1988).

The Marda Fault Zone

The Marda fault zone in the south-Eastern Ethiopia was first recognized in the Marda Range near Jijiga and was called Marda Hills line (Purcell, 1976). The Marda Fault Zone trends northwest/southeast across the Horn of Africa (Morton, 1974; Purcell, 1976) and is expressed in the subsurface as major basement uplift. The "linear NW-SE arrangement of basalt capped summits with basaltic plugs" and the associated Bouger anomaly were considered indications of a major "Volcanic-tectonic" lineament. The fault zone was subsequently described as a complex of NW-SE trending faults, down thrown to the NE and possibly extending 200 km into the Ogaden basin. Recent studies have indicated that the fault zone extends over 400 km beyond the Marda range to the Belet Uen area in Somalia (Fig. 2.3).

The Kuran Fault Zone

The Kuran Fracture Zone has similar features with Marda Fault Zone. It is NW-SE trending fault (Fig.2.3).
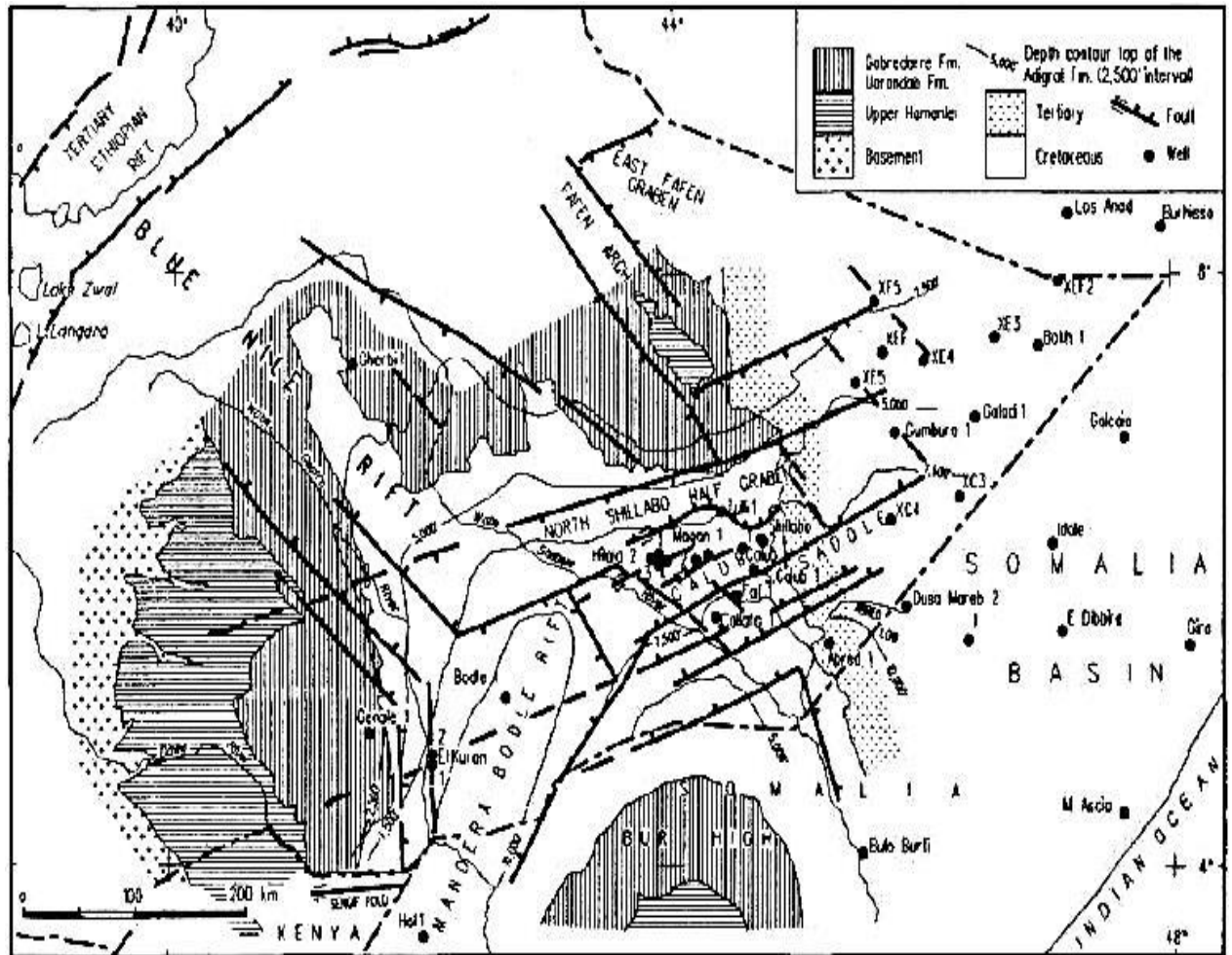
Figure 2.1 Structural map of Ogaden Basin (after Purcell, 1976)
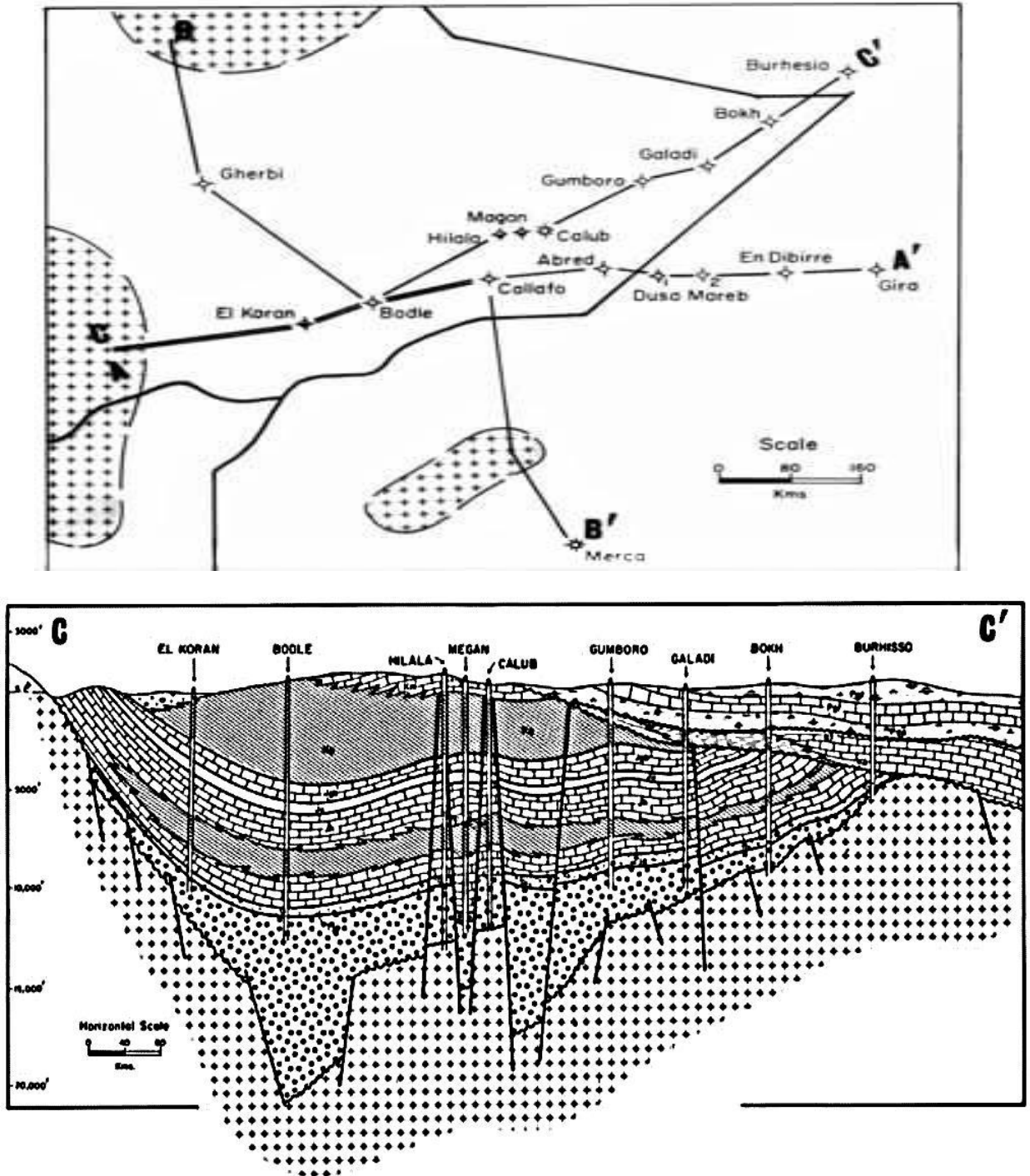
Figure 2.2 C-C'-Northeast-Southwest cross section of Ogaden Basin (after Purcell, 1976)
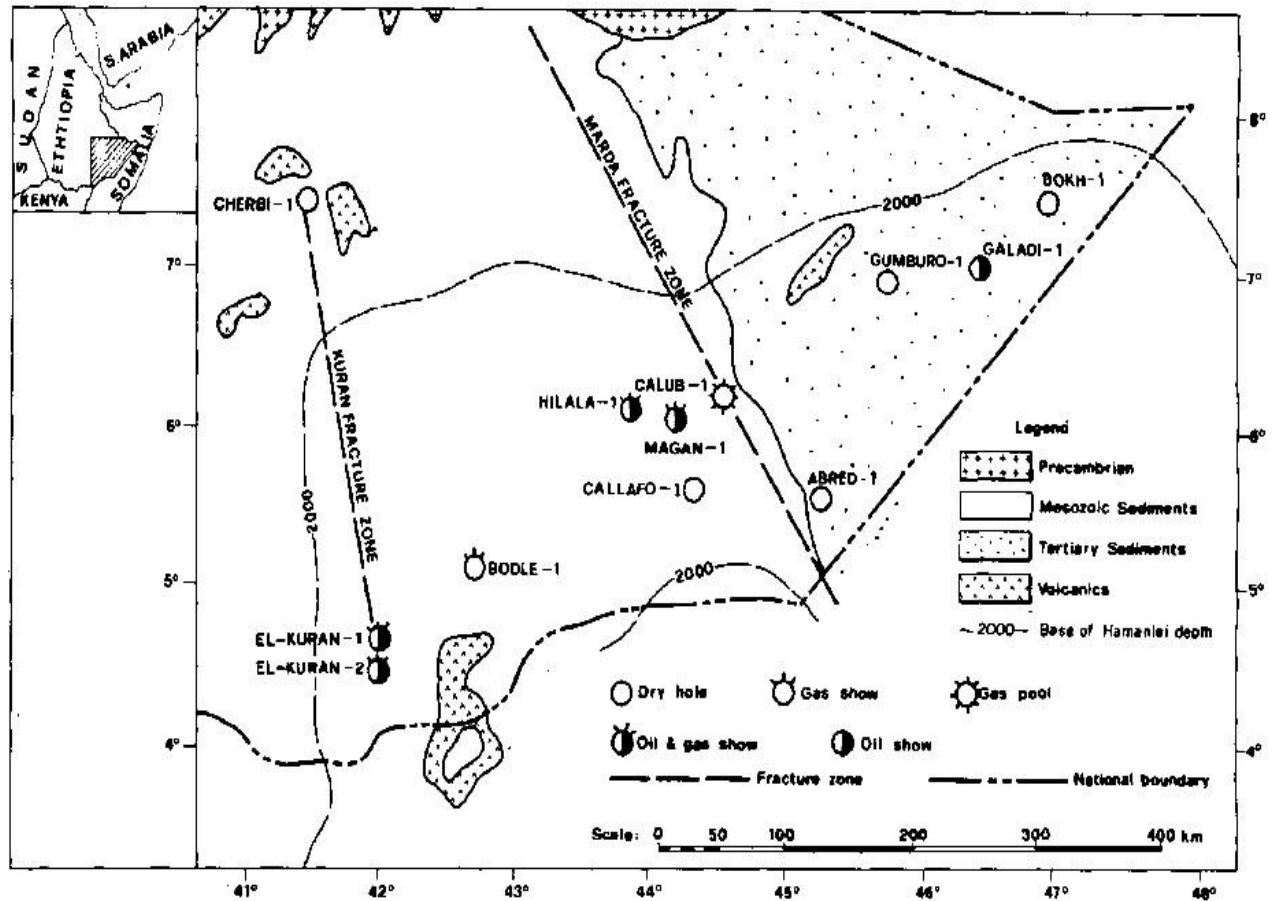
Figure 2.3 map showing generalized surface geology of the Ogaden Basin, test wells and major fracture zones (after Assefa, 1988).

2.3   Stratigraphic succession of Ogaden Basin

The stratigraphic section of Ogaden basin has been established from studies made and drilling by several oil companies and by Ethiopian Institute of Geological Surveys (Worku and Astin, 1992). The following are sedimentary sequences of Ogaden Basin. The general lithostratigraphy of Ogaden Basin is shown in Figure 2.4.

2.3.1   Late Carboniferous to Triassic

BEICIP (1985) as cited in John (2016) and Worku and Astin (1992) concluded the Ethiopian Karoo sediments are subdivided into: **a**. Calub (alluvial fan sandstone; reservoir rock; Late Carboniferous - Permian age) **b**. Bokh (lacustrine shale; source rock; Late Permian / Early Triassic age) **c**. Gumboro (fluvial sandstone; Triassic age). In the Ethiopian Ogaden Basin, maximum formation thicknesses reach 266.5m for the Calub, 445m for the Bokh, and 753m for the Gumburo (BEICIP, 1985).

a.   Calub formation

Assefa (1988), Worku and Astin (1992), and John (2016) indicated the Calub formation rests unconformably on the Precambrian crystalline basement and marks the first sediment filling Ogaden rift valley. It consists of pre-rift sandstones and conglomerates deposited in a glacial-to-fluviatile environment.

2.3.2   Triassic

a.   Bokh formation

The Bokh formation is characterized by organic-rich, black-to-brown shales and laminated sandstones, and corresponds to initial rift-phase deposits laid down in a lacustrine environment. The continental, lacustrine organic shale and silty shale of the Bokh are dominant with minor interbeds of sandstone and dolomite. Contact with over- and underlying formations are conformable and gradual. The black-to-brown shale has a variable organic carbon up to 5% and is locally pyrite rich (Worku and Astin, 1992).

b.   Gumburo formation

The Gumburo formation (middle to upper Triassic) reaches a thickness of 390m in the Calub area and pinches out eastwards. The formation is alluvial, river-deposited sandstone containing abundant land-derived carbonaceous debris and it is mainly the result of sandy braided river deposition, where the major sandy unit was deposited at a fast overall rate of sedimentation from flowing rivers (John, 2016). The formations belong to the Karoo system and vary widely in thickness.

c.   Adigrat sandstone

In the Ogaden Basin, it is thought to overlie the karoo sequence with slight unconformity and it grades upward through a transition zone into the carbonate Hamanlei Formation (Purcell, 1976). It is almost intercepted in all wells in the Ogaden basin and varies in thickness. It comprises fine- to-medium-grained sandstones with intercalated shales, and represents deposition during the early rift phase. According to Shigut (1997) the formation displays the gradational upward change from coarse-to-fine grained and its carbon contents also increases upwards. Its depositional environment is fluviatile succession, braided stream deposit at the base, point bar sequence in the middle, and coastal plain to lagoonal sediments at the top.

d.   Transitional unit

It conformably overlies the Adigrat Formation and underlies the Lower Hamanlei Formation. Shigut (1997) described the unit as it comprises an interbedded sequence of shales,

sandstone, siltstone, silty sandstone, grainy limestones, dolostones and evaporites and its depositional environment from continental to a marine.

2.3.3 Jurassic
a. Hamanlei formation

It represents an Early Jurassic to Callovian syn-rift marine sequence. The Hamanlei formation of Ogaden Basin is subdivided into three lithological units (Shigut, 1997; John, 2016).

i. Lower Hamanlei Formation

It overlies Transitional unit. The formation consists of upward coarsening sequence of shaly limestones and fine-grained to skeletal limestone. The formation is deposited in marine and restricted lagoon environment.

ii. Middle Hamanlei Formation

It conformably overlies Lower Hamanlei and conformably underlies Upper Hamanlei. Middle Hamanlei comprises dolostones, thick evaporite and variety of dolomitized limestones. The shallow inner, locally restricted shelf and a restricted brackish is depositional environment.

iii. Upper Hamanlei Formation

It conformably underlies Urandab Formation and overlies Middle Hamanlei. Upper Hamanlei of the Ogaden Basin is composed of significant thicknesses of oolitic, skeletal grainy limestone abruptly overlying the mixed dolostone and evaporites.

b. Urandab Formation

The formation conformably overlies Upper Hamanlei and underlies Gabredarre Formation. According to Shigut (1997) the formation comprised of dark, laminated marls and limestones containing a pelagic marine fauna, locally gypsiferous and interbedded with shaly mudstone. It represents an open, deep-marine, low-energy environment with slightly anoxic conditions. Purcell (1976) summarized the shale facies predominates over large areas of Ethiopia and Northern Somalia marks the quiet water conditions that prevailed near the peak of the Jurassic transgression. The unit is approximately 400 meters thick.

c. The Gabredarre Formation (Upper Jurassic)

It overlies Urandab Formation. According to Purcell (1976) over most of the central Ogaden Basin, deposition was continuous into the Lower Cretaceous and the contact of the Jurassic

and Cretaceous units is gradational. In the basin areas, the Gabredarre formation consists mainly of grey argillaceous and calcareous marlstones, with interbedded argillaceous limestones and dolomites. Some shale occurs in the upper section, with thin anhydrite layers and occasional fine-grained sandstone. John (2016) concluded the formation represents a gradual shallowing of the sea, resumption of carbonate sedimentation, and the upper disconformity suggests a fall of sea level.

2.3.4   Cretaceous

The opening of the South Atlantic was initiated during the Cretaceous, and a number of shear zones were developed along and within the East African continental margin (Bosworth, 1994). During this post-rift period, regressions and transgressions alternated over the Ogaden platform area and the related sedimentary rocks are represented by the Gorrahei, Mustahil, Ferfer, Belet Uen and Jesomma Formations.

a.   Gorrahei Formation

The type section is also known as Main Gypsum Formation is near Gabredarre in Ethiopia and comprises 200 meters of gypsum intercalated with limestone, marls, and shale (Purcell, 1976). The Main Gypsum Formation outcrops over large areas of the western basin and extends into the Mandera-Lugh trough where it consists of gypsum and limestone with interbedded shales.

b.   Mustahil Formation

The Mustahil Formation consists of alternating limestones and marls with thin shales, rudistid layers and gypsum in the upper part and was deposited in an inner- to the outer-shelf environment. Outcrops of the formation are found in the lower Wabi Shebelli valley and in the center of the western sub-basin (Purcell, 1976; and John, 2016).

Upper Cretaceous sediments outcrop only in the lower Wabi Shebelli valley are subdivided into two formations, the Ferfer Gypsum and the Belet Uen Formation.

c.   Ferfer formation

The Ferfer Gypsum is lithologically similar to the Main Gypsum Formation and consists of alternating dolomites, limestones marls and gypsum. It is approximately 200 meters thick in outcrop and merges by intercalation with the overlying Belet Uen Formation (Purcell, 1976). The facies is believed to represent a restricted lagoonal environment (John, 2016).

d. Belet Uen Formation

It is composed of light-colored limestones, sometimes reefal, with intercalations of grey-green glauconitic shale and green and brown sandstones (Purcell, 1976). Its lower contact is unconformable with older units; however, at its top, it is conformable with the Jessoma Sandstone (John, 2016).

2.3.5 Tertiary

a. Jesomma Sandstone

This Upper Cretaceous-Paleocene sandstone represents a fluvial, shallow- marine depositional environment. Age is based on the age of overlying and underlying sediments. The Jessoma may correlate with beds of the Amba Aradam Sandstone in the Harer area (John, 2016).

b. Auradu Formation

The Auradu Formation is a transgressive fossiliferous mudstone to dolomitic packstone deposited in an inner- to the outer-shelf environment (Purcell, 1976; Bill, 2016).

c. Taleh Formation

The Taleh Formation is lower to middle Eocene evaporitic anhydrite, gypsum, dolomite, cherty limestone, and clay (John, 2016). The Taleh Formation facies indicates a regional shallowing of water depths, partly related to the prograding continental margin but also to regional uplift (Purcell, 1976).

d. Karkar Formation

The Karkar Formation is middle to upper Eocene limestone with marly intercalations and clay (John, 2016). The formation marks a transgressive cycle and is the last marine sequence in the Ethiopian Ogaden area (Purcell, 1976).

e. Trap series

The tertiary section is composed of the thin Palaeocene-Eocene Auradu, Taleh formations in the eastern Ogaden Basin, Oligocene-to-Pliocene Trap Series (volcanic flows) in the northern and western parts of the Ethiopian Plateau, and thicker sedimentary deposits in the Red Sea area to the NE.

2.4   Petroleum system of Ogaden Basin

2.4.1   Potential source rocks

According to Assefa (1988) and Hunegnaw *et al.* (1989) the potential source rocks in the Ogaden Basin include:

a.   Permian-Triassic Bokh Shale

The Bokh Shale comprises black shale, siltstone and silty sandstone with a few dolostone, coarse sandstone and conglomerate intercalations. The Bokh Shale and Hamanlei Formation, contain organic-rich clays, deposited in deltaic, neritic and estuarine environments, thus meet general criteria for oil and gas source.

b.   Transition Zone (top Adigrat-base Lower Hamanlei)

The Hamanlei Formation is a shallow-marine to lagoonal and deltaic deposit that consists of organic-rich carbonate and evaporites with subordinate shale and sandstone.

c.   Upper Jurassic Urandab Shale (Assefa, 1988).

The Urandab Formation is composed of shale, sandstone, marl, cherty and oolitic limestone, as well as dolostone.

2.4.2   Potential reservoir rocks

Drilling in the Ogaden Basin has proved the presence of excellent reservoirs in both the clastic and carbonate units (Purcell, 1976). There are three proven hydrocarbon reservoirs present in the Ogaden Basin (John, 2016). These are Karoo Group Permian age Calub Sandstone, the Upper Triassic- Lower Jurassic Adigrat Sandstone, and the Lower-Middle Jurassic Hamanlei carbonates.

2.4.3   Traps

According to Beicip-Franlab (1985 and 1998) the structural, stratigraphic and combination traps have been identified. Most of the structural traps related to basement tectonics and wrench faulting are present in the central part of the basin, while stratigraphic traps are mainly related to the pinch-out of the Calub Sandstones and are restricted to the margins (Hunegnaw et al., 1998). The traps in the Ogaden Basin are concluded by Hunegnaw *et al.*, as follows:

a. Folded and faulted structures related to transpressional wrench tectonics (drag folds, flower structures along the ENE and NW fault trends);

b. Basement-related features, such as broad, relatively flat domal structures;

c. Fault traps on the flanks of deep grabens;

d. The pinch-out of the Calub Formation reservoir against basement (so far untested by drilling);

e. Deep turbiditic fans within the Calub Formation and in the deepest part of the Shillabo half-graben;

f. Sand lenses, sand bars and channels in the Calub and Adigrat Formations; reefal, oolitic and high-energy facies, as well as zones of dissolution or dolomitization, and zones of local fracturing in the Hamanlei Formation limestones.

2.4.4 Seals

The Calub, Adigrat, Middle and Upper Hamanlei reservoir units are respectively sealed by the Bokh Shales, the Transition Zone, the Middle Hamanlei and Urandab shales, and anhydrite for the Middle Hamanlei seal. These formations have a regional extent, and have an excellent sealing capacity with thicknesses ranging from 30 to 450 m (Hunegnaw *et al*., 1998).

Lithostratigraphy	Depth	Lithology	Depositional enviromnment	Description
Volcanic	0			Volcanic
Clastic				Sandstone, siltstone and shales
Taleh Auradu FM	500		Open marine	Marine ca rbonates and gypsum with m inor am ounts of siltstones
Jessoma sandstone	1000		Fluvial to marginal marine	Fluviatile sandstone with shale intercalation
Belet Uen	1500		Open marine	Marine limestone
Ferfer gypsum	2000		Shallow open marine to restricted lagoonal	Anhydrite, marl and dolomitic lst
Mustahil				Marine limestone
Gorrahei (Main gypsum)	2500 3000 3500 4000		Shallow open marine to restricted lagoonal	Marine limestone, dolomite and shale intercalation
Gebredarre	4500		Shallow open marine	Marine limestone
Urandab			Open marine	Limestone marl and shales
Upper Hamanlei	5000		Shallow open marine	Marine limestone with shale intercalation
Middle Hamanlei	5500 6000		Marginal marine to sabkha	Tidal flat gypsum, limestone, shales and dolomite
Lower Hamanlei	6500		Fluvial to marine	Limestone with shale intercalation
Transition unit			Open marine	Carbonate, shales and Gypsum
Adigrat FM	7000			Sandy conglomerate and sandstone
Gumburo FM	7500		Fluvial	Fluviatile sandstone
Bokh FM	8000		Lacustrine	Lacustrine black shales
Calub	8500		Fluvial	Fluviatile sandstone

Figure 2.4 Stratigraphic section of Ogaden basin (modified from Assefa, 1988 and Wolela, 2008)

CHAPTER 3

3   METHODOLOGY

3.1   Introduction

Right before the initiation of this thesis work, various activities were scheduled. It began with the literature and previous reports reviewing and then followed by problem identification. Next the objectives were set and the methods to be adopted have been selected. After the collection of data and samples, finally the samples were prepared, analyzed and results were interpreted.

3.2   Sample collection

The core cutting samples of Bokh shale from Calub (Calub 2, Calub 3 and Calub 5 wells) utilized for this study were supplied by Ministry of Mines, Petroleum and Natural Gas (MoMPNG) of Ethiopia. A total of twelve shale samples (3 samples from Calub 2; 4 samples from Calub 3; and 5 samples from Calub 5) were collected. The lithological section of wells and sample locations are displayed on the stratigraphic logs produced (Fig 4.1, 4.2 and 4.3).

The samples were then shipped to the laboratory of ALS oil and gas at Houston, Texas for whole rock geochemical analysis. The samples were prepared for three separate analyses: two samples for organic petrography, four samples for XRF elemental analysis, and six samples for TOC and Pyrolysis method.

3.3   Analytical methods

3.3.1   TOC determination and Pyrolysis methods

A total of 6 crushed samples were submitted for analysis. Each of the core samples ground and homogenized using a mortar and pestle prior to analysis. The resulting powders were then prepared for analysis of total organic carbon content and pyrolysis yields.

3.3.1.1 Total organic carbon (TOC) content

Approximately 0.1 grams of the powdered samples were weighed into a porous ceramic leaching crucible and treated with hydrochloric acid to dissolve inorganic matter. After the acid reaction was complete, the crucible was thoroughly rinsed and dried at 105°C for at least 4 hours. The sample remaining in the crucible after leaching was combusted in the LECO induction furnace at a temperature of 1,350°C.

The estimating of TOC was performed using an instrument known as a LECO carbon analyzer to measure TOC values by combusting the organic carbon and measuring the resulting carbon dioxide (CO_2) produced. The percentage of Carbon dioxide generated by the combustion of organic materials in the sample was quantitatively measured using an infrared detector.

3.3.1.2 Pyrolysis method

Programmed Pyrolysis measures the organic richness and thermal maturity of potential source rocks. The procedure is that a geologic sample is introduced into an enclosed vessel and is progressively heated to 650°C under inert gas flow. The total organic content is pyrolyzed in the absence of oxygen, next combusted. The amount of hydrocarbons is monitored by a Flame Ionization Detector (FID). The procedure is completed by oxidation of the residual rock recovered after pyrolysis up to 750°C under oxygen. During pyrolysis S1, S2, and S3 are measured whereas during oxidation S4 and S5 measured while the carbon monoxide and carbon dioxide released are monitored by an infrared cell. Approximately 60 milligrams (mg) of the powdered sample was used in pyrolysis analysis.

The free hydrocarbons, S1 (mg HC/g rock), already present was distilled out of the sample at initial heating of the sample to a temperature of 300°C for 3 minutes. The second peak, S2 (mg HC/g rock) was generated through thermal cracking of non-volatile organic matter when the pyrolysis temperature increased from 300°C to 650°C heating at 25°C/min, while S3 (mg CO2/g rock) corresponds to carbon dioxide released during the same temperature S2 is being generated. The parameters, (HI, mg HC/g TOC) and (OI, mg CO_2/g TOC), used for rock quality assessment are calculated from Pyrolysis data.

3.3.2 Organic petrography/Vitrinite reflectance measurement

All samples were investigated in white- and UV-light using a Zeiss Axio-Scope A1 at 500x (10x eyepiece, and 50x objective) in immersion oil. White- and UV-light was provided by an X-Cite 120 LED light source. Photographs were captured using a Gryphax camera attached to the Zeiss Axio-Scope A1, and Gryphax image-capture software. All images were then reproduced and enhanced in Microsoft PowerPoint software.

3.3.2.1 Sample preparation and analysis

The two shale samples provided for vitrinite reflectance analysis were from Calub 3 and 5 wells. The cutting samples provided were further crushed and embedded in thermoplastic

epoxy in 1.25in molds. The pellets ground and the top surface of the pellets was polished and coated with an immersion oil microscope according to ASTM standards (ASTM, 2011).

Vitrinite reflectance measurements were conducted on both samples. The methodology utilized grey-scale technology that allows for the accurate measurement of small vitrinite particles as small as 4µ or smaller that may be commonly encountered in shales and mudstones as dispersed organic matter. A population of greater than 50 reflectance measurements was taken for each sample.

High-resolution black and white photos were taken with the Gryphax digital camera, and grey-scale values measured using Zeiss AxioScope software. The grey-scale values were then translated into Ro% using a mathematical equation of grey-scale values calibrated to a vitrinite reflectance standard (Schott glass standard with 6 glasses of increasing reflectance). A histogram of all reflectance values collected from an individual sample was then constructed to help identify the in-situ vitrinite population and to subsequently calculate a definitive Ro%.

3.3.3 XRF/Elemental analysis method

XRF analysis provides a bulk elemental composition of rock samples. Four samples of Bokh shale from Calub 3 and Calub 5 (two samples from each well) were analyzed.

3.3.3.1 *Sample Preparation and analysis*

The samples were wet core cuttings which were washed with a mild surfactant to remove any surface contaminants. The cleaned samples are then air dried. Approximately 10-12 grams of the sieved materials using No. 18 mesh (1mm) are subsampled. However, No. 40 mesh (0.4mm) sieve was used when necessary due to sample availability. The sub-samples are subsequently pulverized to a fine powder (<40 microns) using a planetary ball mill. Approximately 4 grams of the dry powdered sample was carefully pressed into pellets using tapered aluminum caps and a hydraulic press. The data were then measured using Bruker S4 Wave-Dispersive XRF instrument.

CHAPTER 4

4   THE LITHOLOGICAL SECTION AND GEOCHEMICAL RESULTS

4.1   Lithological Sections

This study has focused on Calub area in general and the Bokh shale of Calub-2, Calub-3, and Calub-5 Wells in particular. The analytical samples collected from each well are shown below on the lithological sections log (fig. 4.1, 4.2 and 4.3). The Well information is compiled from Soviet Petroleum Exploration and Expedition (SPEE) well technical report and helped the lithological sections produced. The Well data shows that Bokh shale is found at different depth interval and vary in thicknesses in each well. The lithological successions in the Calub wells are presented in Table 4.1. The lithostratigraphy of Calub wells are:

a.   Late carboniferous to Triassic

The sedimentary successions of Upper Paleozoic (Purcell, 1976; Getaneh, 1988) in the study area are:

Calub sandstone: This succession is 34m, 16m, and 185meter thick in Calub 2, 3, and 5 respectively. It is dark-grey argillite with thin layer of light-grey sandstone and silty to sandy texture with no fossil content. It shows bedding structure. This layer overlies basement rock and underlies Bokh shale. It is Permian age sediment (Wolela, 2008; Hunegnaw, 1998; Purcell, 1976).

Bokh shale: It is dark-grey to black argillite and dark mudstone and 393m, 419m, 620 meter thick in Calub 2, 3 and 5 respectively. The Bokh shale is a thinly laminated, uniform and thick unit. The fossils of articulated fish and bivalves have been recorded (Worku and Astin, 1992). This formation underlies Gumburo sandstone and no clear contact between them but gradually coarsening upward. Wolela (2008) and Hunegnaw *et al*. (1998) reported the age of the Bokh shale ranges from Permian to Lower Triassic, i.e. late Paleozoic and early Mesozoic era.

Gumburo sandstone: This formation is an intercalation of light-grey sandstone with thin layer of grey-green argillite, and greyish-green siltstone at the top. It consists of parallel and cross bedded structures. No macrofossils found in this unit. The thicknesses of the units in each section are 22m, 205m, and 48 meter in Calub 2, 3, and 5 respectively. It is the upper unit of Paleozoic formation and underlies Adigrat sandstone of lower Mesozoic formation.

There seems some controversial exists on the age of this formation. Purcell (1976) and Assefa (1988) classified Gumburo sandstone into Permian age whereas Hunegnaw et al. (1998) grouped into Lower Triassic age. Wolela (2008) grouped Gumburo sandstone into lower to middle Triassic age. According to Purcell the absence of a marked unconformity of Gumburo and Adigrat sandstones suggests that deposition was interrupted only briefly hence the Gumburo Sandstone is mainly of Permian age.

b. Late Triassic to Upper Jurassic

Lithostratigraphic units of Mesozoic age in the study area.

Adigrat sandstone: It is light-grey to white sandstone and black, light-green to dark-green argillite interbeds. The dinoflagellates fossil is found at the upper part of this unit. The structures recorded in this unit are from massive to cross bedded. The thickness of the formation is 534m, 317m, and 30 meter in Calub 2, 3 and 5 wells respectively. It underlies transitional series formation and the bottom sequence of Mesozoic era. The age of Adigrat sandstone is from Upper Triassic to Lower Jurassic (Assefa, 1988; Hunegnaw et al., 1998; Purcell, 1976).

Transitional series: It is an intercalation of dark-grey argillite, grey dolomite and thin layer of white gypsum. This unit has dominant bioclasts upward through the succession. It is 44m, 8m, and 90 meter thick in Calub 2, 3, and 5 wells respectively. It is underlain by Adigrat sandstone overlain by Lower Hamanlei.

Lower Hamanlei: It is an interbed of light-grey and dark-grey shaly limestone and dark-grey to black argillite. It is bioturbated by crustacean burrows. The thicknesses of formation in each well are 498m, 335m, and 220 meter in Calub 2, 3, 5 wells respectively. It overlies Transitional series and underlies Middle Hamanlei. Its geological age is in the Lower Jurassic (Hunegnaw et al. 1998; Wolela, 2008; and Worku and Astin, 1992).

Middle Hamanlei: It is an intercalation of brownish-grey dolomite, black argillite, grey gypsum and light-grey limestone with thick deposit of anhydrite and dolomite at the top. This unit is a fossil rich unit notably foraminifers and crustacean coprolites. The succession is 324 meter in Calub 2 well, 244 meter in Calub 3, and 454 meter thick in Calub 5. It overlies Lower Hamanlei and underlies Upper Hamanlei. The geological age of Middle Hamanlei is estimated from Lower to Middle Jurassic (Wolela, 2008).

Upper Hamanlei: It is grey to dark-grey skeletal limestone, light-grey to white anhydrite, and dark and greenish-grey argillite interbeds. The unit thicknesses are 720m, 1094m, and

1076 meter in Calub 2, 3 and 5 wells respectively. It is overlain by Urandab formation and underlain by Middle Hamanlei formation.

Urandab formation: It is dark-grey mudstone, white and grey anhydrite and dark-grey marl. The thickness of the formation in each well is 990m, 813m, and 775 meter in Calub 2, 3 and 5 wells respectively. This formation is fossiliferous containing ammonites, bivalves and brachiopods. It conformably overlies Upper Hamanlei formation and underlies Gabredarre formation. The age of the formation is Middle to Upper Jurassic (Worku and Astin, 1992; Purcell, 1976; Hunegnaw et al. 1998).

Gabredarre formation: It is dark-grey argillite and light-grey to grey limestone and its thickness in the Calub 2, 3, and 5 wells is 170m, 215m, and 75 meter respectively. This is a fossiliferous limestone consisting of bivalves largely and corals. The Gabredarre formation is the upper unit in the study area and it overlies Urandab formation. It is the upper Jurassic formation (Hunegnaw et al. 1998; Assefa, 1988; Wolela, 2008).

Calub-2				Calub-3				Calub-5			
From (m)	To (m)	Formation Name	Lithology description	From (m)	To (m)	Formation Name	Lithology description	From (m)	To (m)	Formation Name	Lithology description
0	170	Gebredarre	Limestone	0	215	Gebredarre formation	Limestone	0	75	Gebredarre	Limestone
170	1160	Urandab	Argillite, anhydrite, dolomite, marl	215	1028	Urandab formation	Argillite, dolomite, anhydrite	75	850	Urandab	Marl, limestone, anhydrite
1160	1880	Upper Hamanlei	Limestone	1028	2122	Upper Hamanlei formation	Limestone, anhydrite, argillite	850	1926	Upper Hamanlei	Limestone, mudstone
1880	2204	Middle Hamanlei	Gypsum, dolomite, anhydrite	2122	2366	Middle Hamanlei formation	Dolomite, anhydrite, gypsum	1926	2380	Middle Hamanlei	Limestone, dolomite, mudstone
2204	2702	Lower Hamanlei	Limestone, argillite	2366	2701	Lower Hamanlei	Limestone, argillite	2380	2600	Lower Hamanlei	Limestone, mudstone
2702	2746	Transitional series	Marl, argillite, limestone	2701	2709	Transitional series	limestone, argillite, gypsum, dolomite	2600	2690	Transitional series	Limestone, mudstone
2746	3280	Adigrat sandstone	Sandstone	2709	3026	Adigrat sandstone	Sandstone, argillite	2690	2720	Adigrat sandstone	Sandstone and mudstone interbedded
3280	3302	Gumburo sandstone	Argillite, siltstone	3026	3231	Gumburo sandstone	Sandstone, argillite, siltstone	2720	2768	Gumburo sandstone	Sandstone
3302	3695	Bokh shale	Argillite	3231	3650	Bokh shale	Argillite	2768	3388	Bokh shale	Mudstone, siltstone
3695	3729	Calub sandstone	Sandstone, argillite	3650	3666	Calub sandstone	Sandstone, argillite	3388	3575	Calub sandstone	Sandstone
3729	3732	Basement	Granite	3666	3690	Basement	Granite	3575	3690	Basement	Granite

Table 4-1 Well data of Calub area (after SPEE, 1986; 1989; 1992)

Calub-2 well: The Bokh formation is 393m thick. The layer consists of mudstone and laminated siltstone and sandstone. Fossils of bivalves and articulated fish are found in this formation. The mudstone is 12m thick and it is dark-grey to black, silty, and hard rock. The sandstone is a grey color, fine-grained, quartzose, dense and hard.
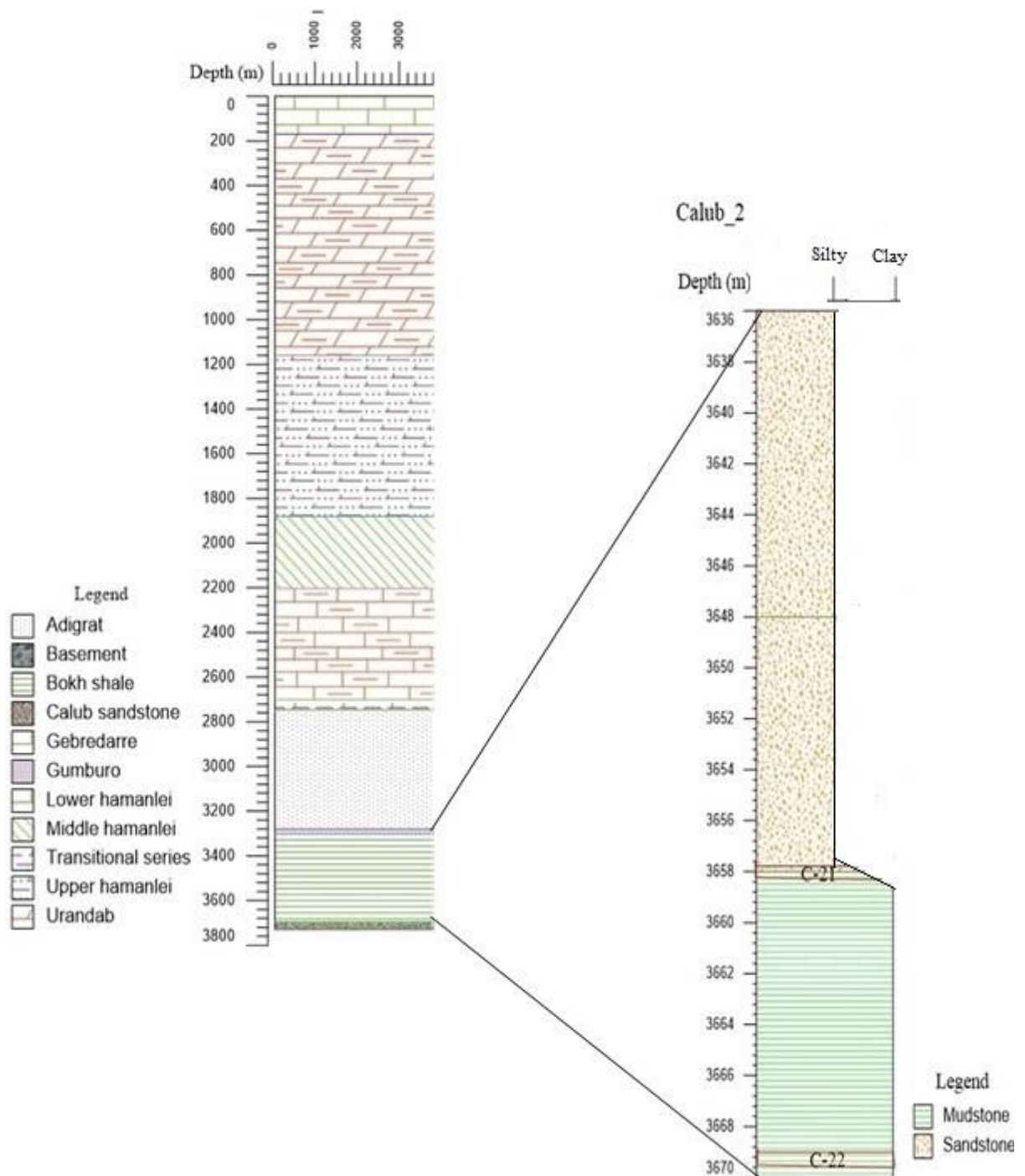
Figure 4.1 A. Generalized section of Calub-2 of Ogaden Basin. B. Stratigraphic section of Bokh Formation

Calub-3 well: The Bokh formation is 419m thick. It is a layer of laminated argillite with siltstone. Fossils of bivalves are found in the Bokh shale. The argillite is grey to dark-grey, non-calcareous and friable, medium hard.
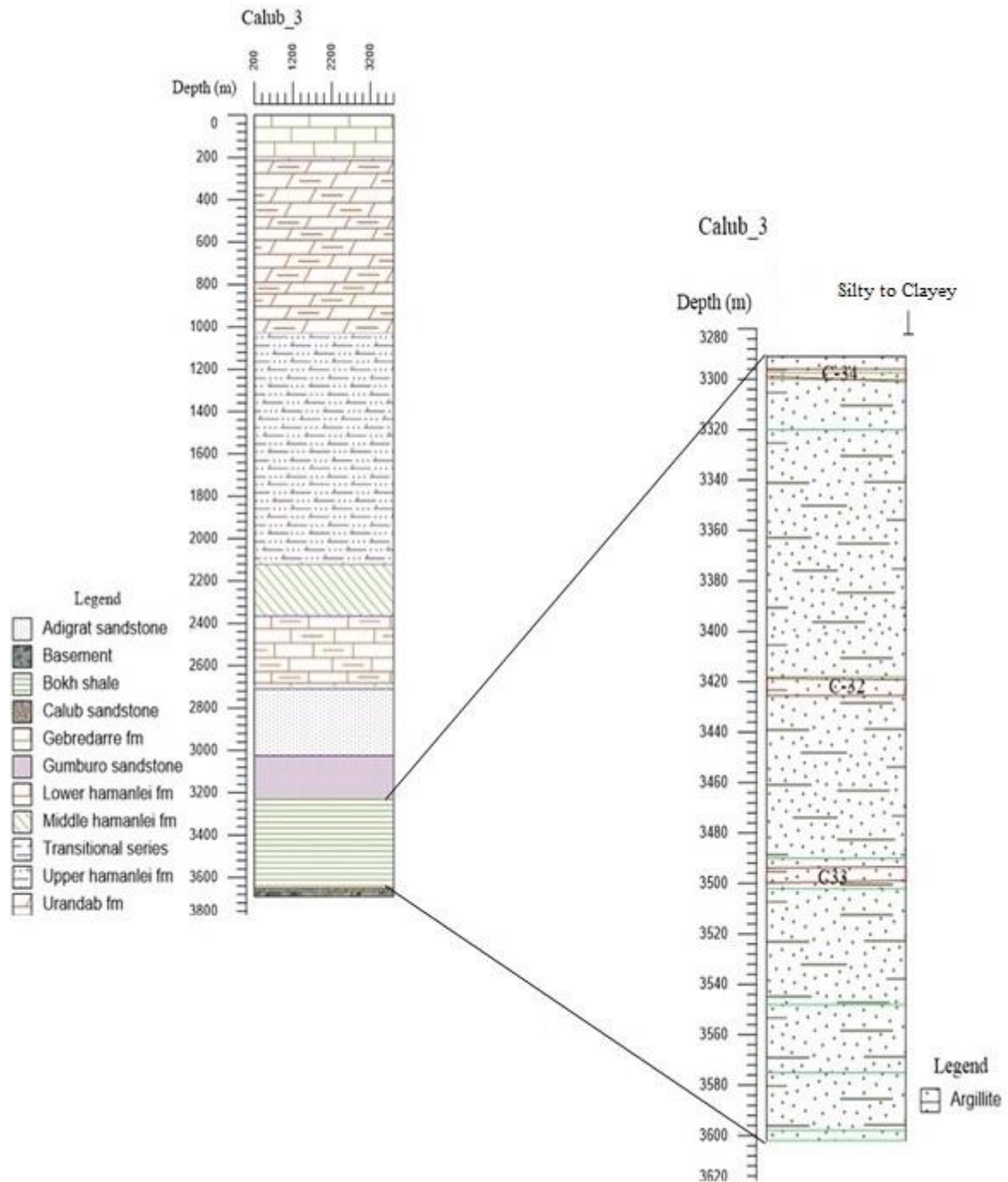
Figure 4.2 A. Generalized section of Calub-3 of Ogaden Basin. B. Section of Bokh Formation

Calub-5 well: The Bokh formation in this well is 520m thick. The layer is formed from lamination of mudstone, siltstone, and sandstone with no fossil content. The mudstone is 20m thick and it is dark-grey to black, brittle and fractured, horizontally bedded sediment. The sandstone is light-grey, medium to coarse-grained intercalated with dark-grey silty mudstone. The siltstone is grey-brown, fine-grained sandstone and siltstone intercalation.
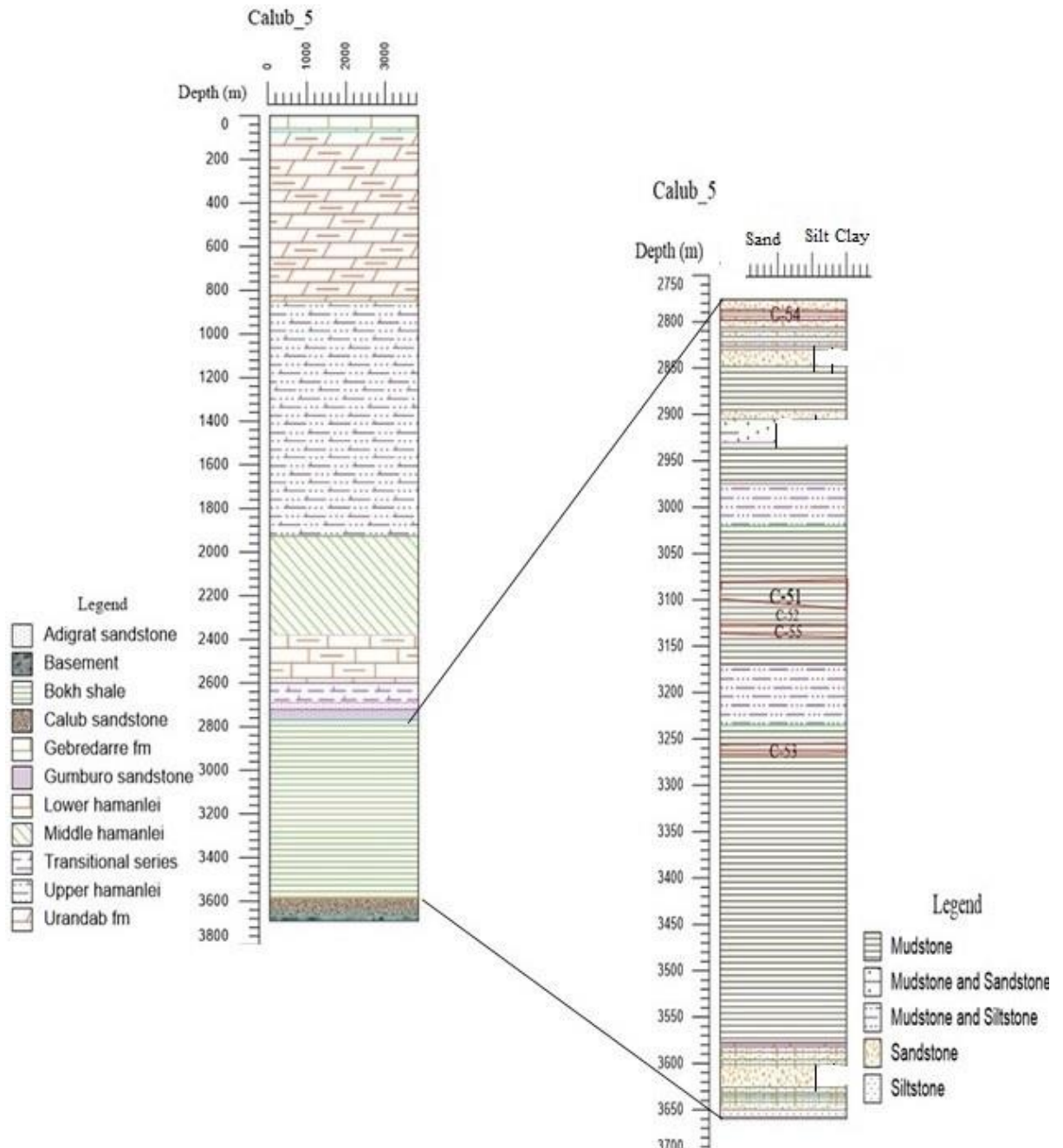
Figure 4.3 A. Generalized section of Calub-5 of Ogaden Basin B. Stratigraphic section of Bokh Formation

4.1.1   Correlation of studied lithological section

The thickness and depth of Bokh shale and their lateral successions are shown below. The orientation of the lithological logs is from West to East.
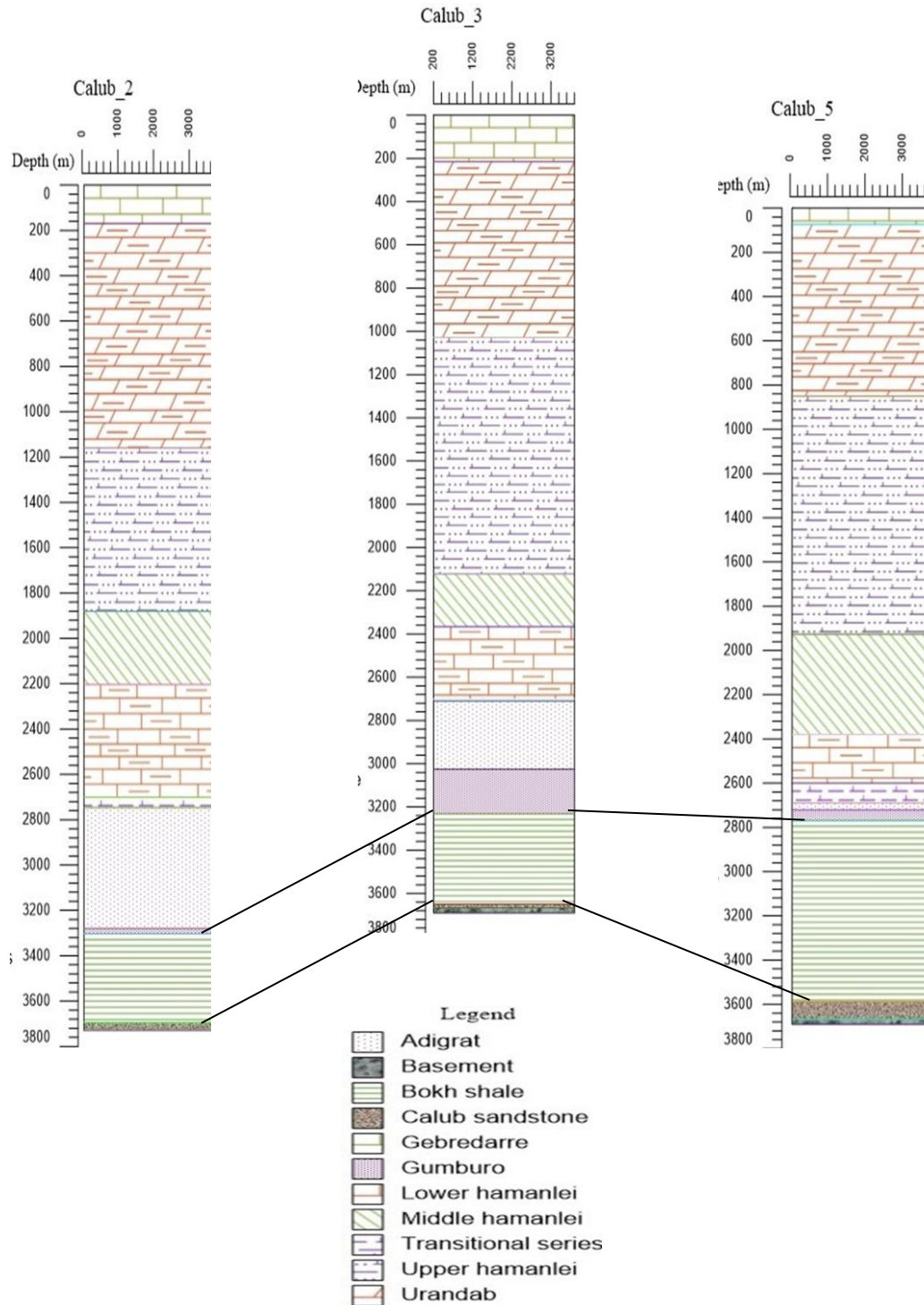
Figure 4.4 Correlation of Stratigraphic sections of Bokh shale in the Calub area.

4.2   Geochemical Results

The formation intervals from where the samples were collected for geochemical and organic petrographic analysis from all the three wells are presented (*Table 4.1 and Fig. 4.1, 4.2 and 4.3*). The total of three samples from Calub 2 at depths of 3624m, 3635m, and 3660m were taken and analyzed for TOC and pyrolysis. Four samples from Calub 3 at depths of 3221m, 3298m, 3420m, and 3495m were taken and analyzed for three separate methods, and five samples at depths of 2771m, 2851m, 3092m, 3133m, and 3257m were taken from Calub 5 and prepared for Vitrinite reflectance, XRF elemental analysis, and TOC and Pyrolysis method analyses.

4.2.1   TOC and Pyrolysis results

Five shale samples, C-21 and C-22 from Calub 2 at depth of 3659m and 3668 meter respectively (*Fig. 4.1*); C-34 from Calub 3 at depth of 3298m (*Fig. 4.2*); C-54 and C-55 from Calub 5 at depth of 2771m and 3133 meter respectively (*Fig. 4.3*), were taken and analyzed for TOC and Pyrolysis methods. Eventually two parameters, the essential rock-eval (measured) and the calculation of parameters was done at the laboratory.

Measured parameters are pyrolysis data, i.e. TOC, S1, S2, S3, and Tmax whereas calculated parameters: are values calculated from pyrolysis data.

No.	Well name	Sample Id	Depth (m)	TOC	Free Hydrocarbons	Generatable Hydrocarbons	Temp of Peak S2	Generatable CO_2	HI	OI	GP
				%	S1 (mg HC/g rock)	S2 (mg HC/g rock)	Tmax (^{o}C)	S3 (mg CO2/g rock)	mgHC/g TOC	mgCO2/g TOC	mg HC/g rock
1	Calub_2	C-21	3659	0.56	0.07	0.12	446	0.45	21.00	80.00	0.19
2	Calub_2	C-22	3668	0.52	0.23	0.27	338	0.29	52.00	56.00	0.5
3	Calub_3	C-34	3298	0.50	0.03	0.06	457	0.25	12.00	50.00	0.09
4	Calub_3	C-35	3543	0.37	-	-	-	-	-	-	-
5	Calub_5	C-54	2771	0.53	0.03	0.08	449	0.26	15.00	50.00	0.11
6	Calub_5	C-55	3133	0.49	0.02	0.05	458	0.19	10.00	39.00	0.07

Note:

HI = S2*100/TOC, **OI** = S3*100/TOC, **PI** = S1/ (S1+S2), and **GP** = S1+S2

Table 4-2 TOC and Pyrolysis results

4.2.2   Vitrinite reflectance result

Two shale samples, C33 from Calub 3 at depth 3495m; and C53 from Calub 5 at depth 3257m, were examined for vitrinite reflectance analyses.

a.   Sample C33

The sample C33 exhibits a Ro (random) 1.79%, with a calculated Ro (max) 1.89%. The standard deviation of the interpreted vitrinite reflectance measurements is 0.192 with a sample population of 53 (Table 4.3A).

b.   Sample C53

The sample C53 exhibits a Ro (random) 1.30%, with a calculated Ro (max) 1.37%. The standard deviation of the interpreted vitrinite reflectance measurements is 0.354 with a sample population of 52 (Table 4.3 B).

C33									
1.04	1.42	1.45	1.46	1.50	1.50	1.56	1.56	1.58	1.59
1.63	1.68	1.69	1.69	1.70	1.73	1.73	1.74	1.74	1.75
1.75	1.78	1.81	1.82	1.82	1.82	1.82	1.83	1.84	1.88
1.88	1.89	1.90	1.91	1.91	1.93	1.93	1.93	1.94	1.94
1.94	1.94	1.94	1.96	1.96	1.96	1.96	1.96	1.97	1.97
1.98	1.99	1.99							

Table 4.3 A: *Tabular representation of raw Data of Vitrinite-Reflectance for C33*

C53									
0.50	0.91	0.96	0.99	1.09	1.09	1.11	1.12	1.12	1.18
1.20	1.21	1.23	1.23	1.25	1.26	1.27	1.29	1.30	1.31
1.32	1.34	1.37	1.38	1.39	1.45	1.46	1.46	1.46	1.46
1.46	1.48	1.52	1.53	1.60	1.62	1.77	1.83	1.91	1.93
1.93	1.94	1.94	1.96	1.96	1.98	1.98	1.98	1.98	1.99
1.99	1.99								

Table 4.3 B: *Tabular representation of raw Data of Vitrinite-Reflectance for C53*

Table 4-3 Numerical Vitrinite reflectance results
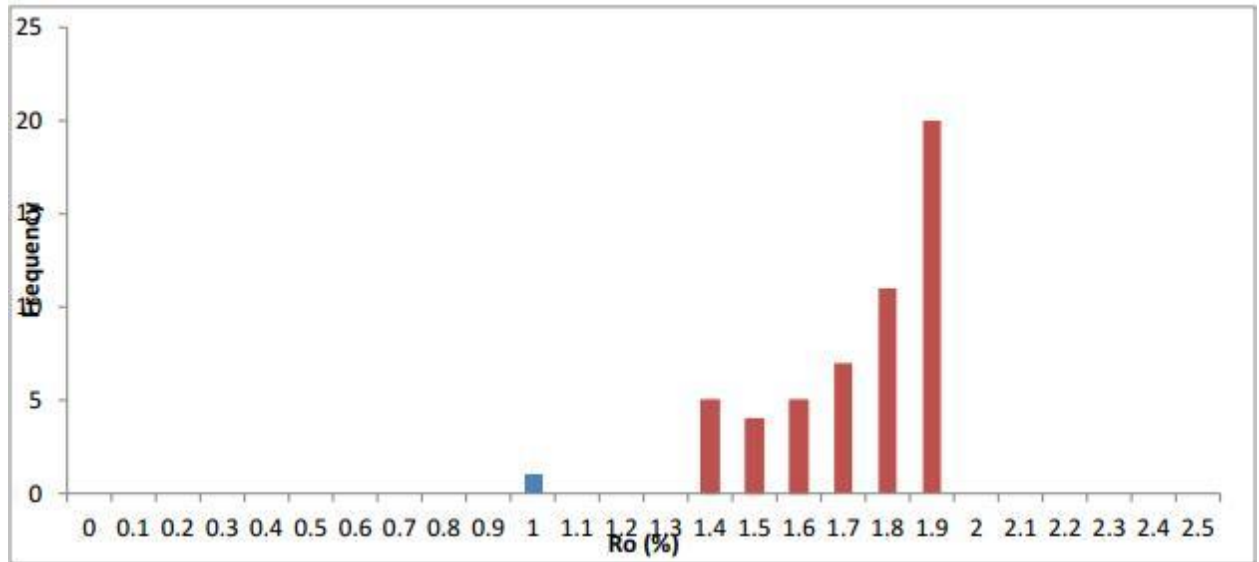
Figure 4.5A. *The samples in Red (Middle set) are interpreted as in-situ vitrinite, whereas those values in Blue are interpreted as lower reflecting solid bitumen.*

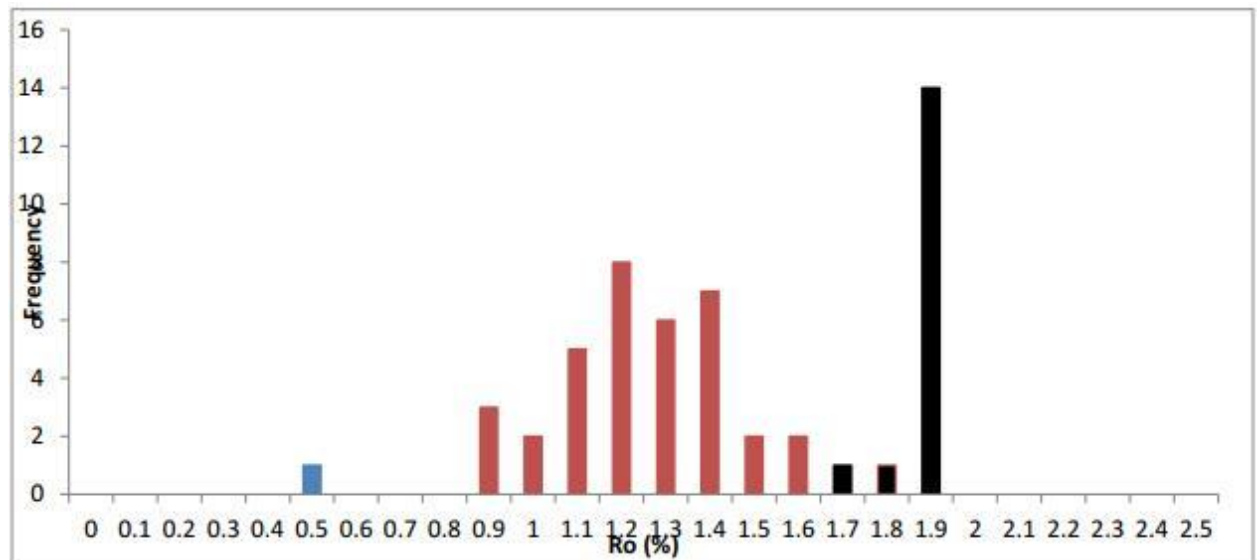**Figure 4.5 B**. *The samples in Red are interpreted as in-situ vitrinite, those values in Blue are interpreted as lower reflecting solid bitumen, and those values in Black are interpreted as high-reflecting inertinite macerals*

Figure 4.5 A. Histogram showing the distribution of vitrinite reflectance values collected from the sample C33 B. Histogram showing the distribution of vitrinite reflectance values collected from the sample C53.

4.2.3 XRF Result

Four shale samples, C31 and C32 from Calub 3 at the depth of 3221m and 3420m respectively; and C51 and C52 from Calub 5 at the depth of 3092m and 3133m respectively are collected and examined for their elemental. The following is the result of XRF elemental analysis (*Table 4.4 A and B*).

Sample Name	Al_2O_3 %	SiO_2 %	TiO_2 %	Fe_2O_3 %	MnO %	MgO %	CaO %	Na_2O %	K_2O %	P_2O_5 %
C31	18.2	58.0	0.9	7.1	0.1	2.4	0.4	0.7	4.7	0.2
C32	17.7	58.6	0.8	6.9	0.1	2.4	0.5	0.7	4.7	0.2
C51	16.9	57.4	0.8	7.1	0.1	2.6	2.1	0.9	3.3	0.2
C52	17.5	59.7	0.8	7.2	0.1	2.4	0.4	1.0	3.4	0.1

Table 4.4 A. *Major and Minor Elements*

Sample Name	S Ppm	Cl Ppm	As Ppm	Ba Ppm	Ce Ppm	Co Ppm	Cr ppm	Cs Ppm	Cu Ppm	Ga ppm	Hf ppm	La ppm
C31	1174	408	0	360	120	18	86	9	30	22	6	61
C32	1201	410	0	429	111	17	82	7	26	22	7	54
C51	8186	403	3	367	95	17	83	12	29	20	7	50
C52	7617	404	2	488	98	19	84	10	27	21	7	51

Mo Ppm	Nb ppm	Ni Ppm	Pb Ppm	Rb ppm	Sc Ppm	Sr ppm	Ta Ppm	Th Ppm	U ppm	V Ppm	Y Ppm	Zn ppm	Zr ppm
0	18	40	22	166	14	125	0	16	5	113	36	71	207
1	17	40	22	160	14	122	0	15	5	104	33	68	218
4	18	41	28	133	14	135	0	13	4	132	29	84	196
6	17	43	27	138	13	95	0	13	5	138	30	85	207

Table 4.4 B. *Trace Elements*

Table 4-4 Geochemical results of major, minor and trace elements

CHAPTER 5

5   DISCUSSION

5.1   Introduction

The organic geochemical analysis provides analytical data to the identification of the richness of source rock, type of organic matter, and thermal maturity of source rocks (Peters and Cassa, 1994). Rock-Eval Pyrolysis technique has been widely used to evaluate the hydrocarbon generation potential, organic matter type, maturity of the shale rock (Espitalie, 1986; and Peters, 1986). These geochemical requirements are interpreted from Pyrolysis data and calculated parameters. The methods of source rock assessment were carried out by both direct and indirect ways. Of the studied shale five samples were directly (rock-eval) analyzed and two samples examined indirect (vitrinite reflectance). In addition, inorganic geochemical analysis of 4 shale samples conducted for depositional environment interpretation and provenance characterization. The overall results are *presented in Chapter 4* and it is discussed vis-à-vis standard guidelines by various workers shown in table 5.1(Espitalié*et al.,* 1984; Peters K.E., 1986; Peters K.E. and Cassa, M.R., 1994).

Quantity			
	TOC (wt %)	S1(mg HC/g rock)	S2(mg HC/g rock)
Poor	<0.5	<0.5	<2.5
Fair	0.5-1	0.5-1	2.5-5
Good	1-2	1-2	5-10
Very good	2-4	2-4	10-20
Excellent	>4	>4	>20
Quality			
	HI (mg HC/g rock)	S2/S3	Kerogen type
None	<50	<1	IV
Gas	50-200	1-5	III
Gas and oil	200-300	5-10	II/III
Oil	300-600	10-15	II
Oil	>600	>15	I
Maturity			
	Ro (%)	Tmax (^{o}C)	
Immature	0.2-0.6	<435	
Early Mature	0.6-0.65	435-445	
Peak Mature	0.65-0.9	445-450	
Late Mature	0.9-1.35	450-470	
Post Mature	>1.35	>470	

Table 5-1 Interpretation guidelines for source rock evaluation (Espitalié et al., 1984; Peters K.E., 1986; Peters K.E. and Cassa, M.R., 1994)

5.2   Discussion

The geochemical results of the total organic carbon (TOC) and Rock-Eval pyrolysis of the Bokh shale are presented in *Table 4.2 A* and *B* and that of vitrinite reflectance is in *Table 4.3 A and B*. The essential parameters of Rock-Eval that were measured directly are: TOC; S_1, free hydrocarbon; S_2, pyrolyzed hydrocarbon from cracking of kerogen; S3, the quantity of CO_2; and Tmax while the calculated parameters are Hydrogen Index (HI), Oxygen Index (OI), Generative Potential (GP), and Productivity Index (PI). Then, both measured and calculated parameters were cross-plotted in order to determine the geochemical requirements, i.e. quantity, quality, and thermal maturity of organic matter contained in the shales. Finally, the plots were interpreted to the evaluation of the potential of source rocks.

5.2.1   Organic Richness and hydrocarbon potential

The quantity of organic matter is one of the working parameters in the evaluation of petroleum source rocks. The organic richness of source rock is determined from measured TOC. Tissot and Welte (1984) described TOC is a bulk sedimentary parameter that represents the fraction of organic matter that has survived degradation and early diagenesis in the sediments. Total organic carbon is the quantity of organic carbon in a rock sample and includes both kerogen and bitumen (Peters and Cassa, 1994).

5.2.1.1   Organic richness

Total organic carbon (TOC, wt. *%)* describes the quantity of organic carbon in a rock sample and includes both kerogen and bitumen (Peters and Cassa, 1994). The TOC data from the pyrolysis of Bokh shale ranges from 0.495 to 0.56% with an average of about 0.521wt% and the free S_1 and generatable S_2 hydrocarbons ranges from 0.02 to 0.23 mgHC/g rock and 0.05 to 0.27 mg HC/g rock respectively (Table 4.2 A). The variation in TOC content is caused due to climatic variations of sediment deposition, decomposition, weathering effect and/ or thermal maturation with burial (Hunt, 1995; Stein, 2007; Wang and Carr, 2013). The TOC of one sample from Calub 3 at depth of 3543m is 0.37wt% and it was rejected while interpretation since it is anomalous. The average TOC value of the samples is nearly closer to the background value (0.5 %) of petroleum potential source rocks and S_1 and S_2 are less than the minimum required (0.5 mgHC/g rock for S_1 and 2.5 mg HC/g rock for S_2). S_2 is a more realistic measure of source rock potential than TOC since TOC includes "dead carbon" incapable of generating petroleum (Peters and Cassa, 1994). The previous reports by

Hunegnaw et al (1998) and Wolela (2008) indicated the TOC ranging from 0.5 to 1.5% and Worku and Astin (1992) suggested the organic content of Bokh shale ranging up to 5%.

5.2.1.2 Hydrocarbon potential

A. Genetic Potential (S_1+S_2): is the total amount of petroleum that the kerogen is able to generate if it is subjected to an adequate temperature during a sufficient interval of time (Tissot and Welte, 1984). According to Tissot and Welte (1984), and Hunt (1996) source rocks with a GP<2, from 2 to 5, from 5 to 10 and >10 are considered to have poor, fair, good, and very good generation potential, respectively. The average GP of the studied samples is 0.192 mg HC/g rock varying from 0.07 to 0.5 mg HC/g rock. The overall GP of the shale sample is less than 2 that indicates poor generation potential (*Table 5.2*).

Bore hole	GP (mg HC/g TOC)			Evaluation
	Min	Max	Avg.	
Calub_2	0.19	0.5	0.345	
Calub_3	0.09	0.09	0.09	Poor
Calub_5	0.07	0.11	0.09	

Table 5-2 generation potential of Bokh shale

B. Productivity Index (PI): production index or transformation ratio, is a proportionality between the hydrocarbons that already generated (S1) from a kerogen and quantity of whole hydrocarbons that can be obtained from kerogen. The production index or productivity index (PI) is derived from the relationship S1/ (S1+S2). The PI of studied samples shows a range of 0.27 to 0.46 mgHC/g TOC and indicates insitu petroleum generation (Peters & Cassa, 1994) of matured sediments except C-22, with 0.46 mgHC/g TOC and suggesting sample contamination by well additives (Peters, 1986). The studied Bokh shale is poor in organic matter constituent and proves indigenous petroleum generation.

5.2.2 Organic matter type

In addition to organic richness, it is important to identify the quality of organic matter as it influences the hydrocarbon product. The quantity and maceral composition of kerogen determine petroleum potential and can differ vertically or laterally within a source rock. According to Peters (1986) and Peters and Cassa (1994), kerogen types in sedimentary rocks are: Type-I kerogen is composed of oil-prone hydrogen-rich organic matter generally in lacustrine and some marine sediment whereas Type II comprising oil prone hydrogen-rich organic matter of marine sediments and also generates gas. Type III kerogen is gas prone, terrestrial organic matter derived mainly from woody plant material that is low in hydrogen

contents while Type IV kerogens contain very little hydrogen (inert). The type of organic matter was identified from HI and S2, and cross-plots of HI *versus* OI and TOC *versus* S2.

5.2.2.1 HI and S2/S3

HI and S2/S3 are proportional to the amount of hydrogen in the kerogen and thus indicate the potential of the rock to generate oil (Peters and Cassa, 1994). Low hydrogen indices indicate less potential to generate oil, and HI and S2/S3 less than 50 and 1 respectively indicates type IV kerogen (inert). These kerogens are dominated by inertinite macerals that generate little or no hydrocarbons during maturation. The studied samples HI are from 10 to 52 mgHC/g TOC with an average of about 22 mgHC/g TOC whereas; S2/S3 is from 0.24 to 0.93 with an average of about 0.4. Both parameters indicate the studied samples are genetically type IV kerogen. Therefore, the source rock is assumed to be inertinite dominated macerals.

5.2.2.2 Oxygen index (OI) versus Hydrogen index (HI)

Espitalie et al (1984) showed that oxygen (OI) in the kerogen is proportional to the carbon dioxide liberated during pyrolysis (S3) and that the hydrogen content (HI) is proportional to the hydrocarbons liberated (S2). They defined the hydrogen index (HI) vs. oxygen index (OI) plot where HI and OI are (S2/TOC) x 100 and (S3/TOC) x 100, respectively. HI versus OI plots are generally reliable indicators of kerogen type. It is indicated that the samples of Bokh shale have the HI ranging from 10 to 52 mg HC/g TOC with an average HI of 22 mg HC/ g TOC. Type IV kerogen commonly has HI values of <50 mg HC/g TOC (Tissot and Welte, 1984; Peters and Cassa, 1994). OI value ranges from 39 to 80 mg CO_2/g TOC with an average of 55 mg CO_2/g TOC. The cross plot between HI and OI of samples (Figure 5.1) denotes the presence of type IV, inert (Hunt, 1995). However, Hunegnaw *et al*. (1998) reported that the Bokh Shale contain Type II organic matter, mixed with minor Type III.
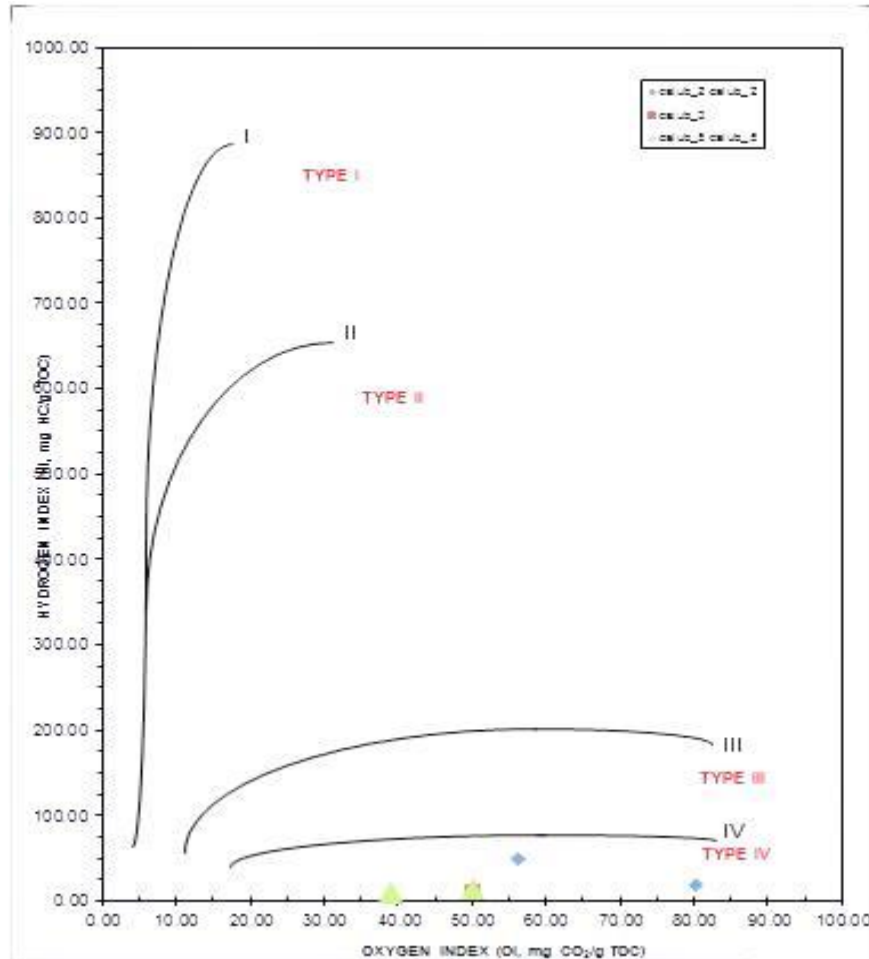
Figure 5.1 OI vs. HI plot of Bokh shale (after Van Krevelen, 1961)

5.2.2.3 TOC versus S2

In the guidelines Table 5.1 it is indicated that samples which contain TOC less than 0.5 wt % and S2 less than 2.5 mg/g are considered poor source rocks. Samples contain from 0.5 to 1.0 wt % TOC and S2 from 2.5 to 5 mg/g are fair source rocks and those containing TOC from 1-2 wt % and S2 from 5-10 mg/g are good source rocks and samples that contain more than 2 wt% TOC and S2>10 mg/g are considered very good source rocks. The plot of TOC versus pyrolysis S2 was interpreted to determine the type of organic matter (*Figure 5.2*). The analyzed sample shows TOC ranging between 0.495 and 0.56 wt% and S2 ranging from 0.05 to 0.27 mg HC/g rock. Figure 5.2 illustrates that the samples are of type IV kerogen.
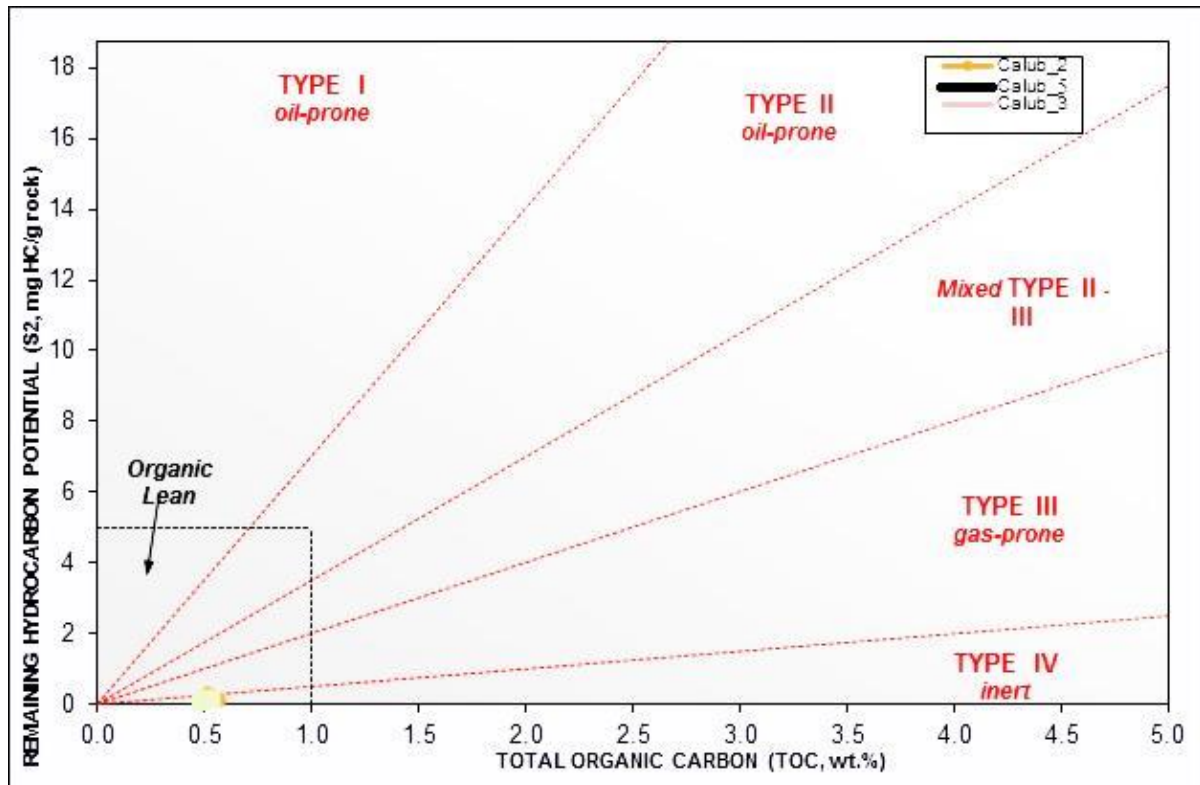
Figure 5.2 TOC vs. S2 cross plot of Bokh shale (after Langford and BlancValleron, 1990)

5.2.3   Thermal maturity determination

Thermal maturity refers to the extent of temperature-time driven reactions that convert sedimentary organic matter (source rock) to oil, wet gas, and finally to dry gas and pyrobitumen (Peters and Cassa, 1994). The degree of thermal alteration of organic matter due to heating provides an indication of source rock maturity. The degree of evolution of the organic matter is commonly expressed in terms of vitrinite reflectance. The evaluation of thermal maturity of the organic matter by optical microscopy is based on visual assessment (e.g., spore coloration) or direct measurement of optical properties (e.g., huminite/vitrinite reflectance measurement).

The thermal maturity of source rock can be estimated from Tmax, vitrinite reflectance, and Tmax versus PI graph.

5.2.3.1 Tmax for thermal maturity level

Organic matter has three different maturity phases depending on degree of thermal alteration (Espitalie et al., 1984; Peters, 1986; Peters and Cassa, 1994).   Immature source rock (Tmax<435^{0}C), which has not been obviously affected by temperature and may be affected by biological diagenesis processes whereas, Mature (435-470^{0}C), which is (or was), within an

oil window and has been converted to petroleum via thermal processes/ catagenesis processes; and Post-mature (Tmax>470^{0}C), which is in the gas window because it is hydrogen deficient material due to the influence of high temperatures/metagenesis processes (Table 5.1).

The analyzed samples, C-21, C-34, C-54 and C-55, Tmax range from 446°C to 458°C, except one sample from Calub-2 (C-22) is 338°C. This range confirms that the studied samples are mature whereas C-22 is immature.

5.2.3.2 Vitrinite reflectance

The purpose of the vitrinite reflectance analysis was to understand maturation information of the source rock and Bokh shale in this case. Two samples one from Calub-3 (C-33) from depth of 3495m and another sample from Calub-5 (C-53) at depth of 3257m were analyzed for their reflectance response. The analytical results of each sample are given (Table 4.3A and B).

Vitrinite is a type of kerogen particle formed from humic gels thought to be derived from the lignin-cellulose cell walls of higher plants (Teichmuller, 1989). Vitrinite reflectance measured in incident white light under oil immersion is the most robust parameter used in organic petrology to define the level of maturity of sedimentary rocks containing dispersed organic matter (ICCP, 1971; Stach et al., 1982; Taylor et al., 1998).

Thermally immature source rocks have been affected by diagenesis without a pronounced effect of temperature (<0.6% Ro) and are where microbial gas is produced. Thermally mature (0.6-1.35) organic matter is in the oil window has been affected by thermal processes covering the temperature range that generates oil (0.6-1.0% Ro). Thermally post-mature (>1.35% Ro) organic matter is in the wet (1- 1.35%Ro) and dry gas zones (1.35-3% Ro) that it has been reduced to a hydrogen-poor residue capable of generating only small amounts of hydrocarbon gases (Dow, 1977; Senftle and Landis, 1991; Peters, K.E., and Cassa M.R.1994; Botoucharov, 2007).

Calub-3: Ro histograms show the frequency distribution of reflectance measurements determined on about 50-100 vitrinite particles in each polished kerogen preparation because Ro values based on fewer than 50 particles can be unreliable (Peters and Cassa, 1994).

The studied sample, C-33, shows the highest histogram peaks 1.4% to 1.9 Ro % (Fig.4.5 A) are interpreted as being insitu vitrinite. There is a relatively low standard deviation among the

insitu vitrinite reflectance values. It should be noted that insitu organic matter is sparse in this sample, and collecting the requisite 50 data points was extremely difficult, owing to the spread of Ro values; identifying insitu vitrinite in comparison to other observable organic matter (e.g. solid bitumen and semi-fusinite) was quite difficult. Vitrinite reflectance (Ro) increases during thermal maturation due to complex, irreversible aromatization reactions (Peters and Cassa, 1994).

The analyzed sample exhibits a random 1.79 Ro %, with a calculated Ro (max) of 1.89%. The standard deviation of the interpreted vitrinite reflectance measurements is 0.192 with the sample population of 53. The value is coincident with the interval of the late wet-gas to dry-gas hydrocarbon window. This high reflectance is corroborated by a distinct lack of organic matter fluorescence. The lack of observable fluorescence corroborates a high maturity (over mature).

Calub-5: The analyzed sample, C-53, indicates the middle histogram peaks 0.9% to 1.6 Ro % (Fig.4.5 B) are interpreted as being insitu vitrinite. There is a small population of measured low-reflecting solid bitumen and a relatively high number of higher-reflecting inertinite measurements. The high standard deviation indicates a wide spread of Ro values, and the cluster of values from 1.1 to 1.4 Ro % could be interpreted as a better representation of insitu vitrinite. This would slightly shift the Ro (random) to be 1.31% with a Ro (max calculated) at 1.39%, since mean random Ro is determined from histogram and it is slightly higher than the current interpreted value. The extremely weak to non-existent fluorescence would indicate high maturity, in the late oil window (wet-gas) or beyond (dry-gas).

The sample C53 exhibits a random 1.30 Ro % and the calculated Ro (max) of 1.37%. The standard deviation of the interpreted vitrinite reflectance measurements is 0.354 with a sample population of 52. This reflectance value agrees with the intervals of late mature and may be in the wet-gas hydrocarbon window. This reflectance is corroborated by very poor and weak fluorescence.

Therefore from the vitrinite reflectance data thermal maturity of the studied samples range from late mature to over-mature. Previous work by Hunegnaw *et al.* (1998) shows the calculated vitrinite reflectance vary between 1 to 1.3 Ro.

5.2.3.3 Tmax versus PI

The Cross plot and relations between the essential Rock-Eval parameter, Tmax, and the calculated Rock-Eval parameter, PI, is an important method for indicating the thermal

maturity of organic matter. Peters (1986) and Peters and Cassa (1994) applied Tmax and PI geochemical parameters to estimate thermal maturity level. Accordingly, immature organic matter has Tmax and PI values less than 430°C and 0.10, respectively. The PI reaches about 0.4 (at the bottom of the oil window or beginning of the wet-gas zone) from 0.1 for mature organic matter and increases to 1.0 when the hydrocarbon-generative capacity of the kerogen has been exhausted. A Tmax greater than 470°C represents the wet-gas zone.

Most of the studied samples have T$_{max}$> 435 and PI of 0.27 to 0.37. Two samples, one from Calub-2 have 0.37 PI at 446°C and another from Calub-5 have 0.27 PI at 449°C, are mature source rock in oil window which underwent intensive oil generation and expulsion. The other two samples from Calub-3 have 0.33 PI at 457°C and Calub-5 have 0.285 PI at 458°C falls within condensate-wet gas zone indicating capable of generating wet-gas. But, one of the samples from Calub-2 has PI of 0.46 at Tmax 338°C which is immature and suggests contaminated source rock (Fig 5.3).
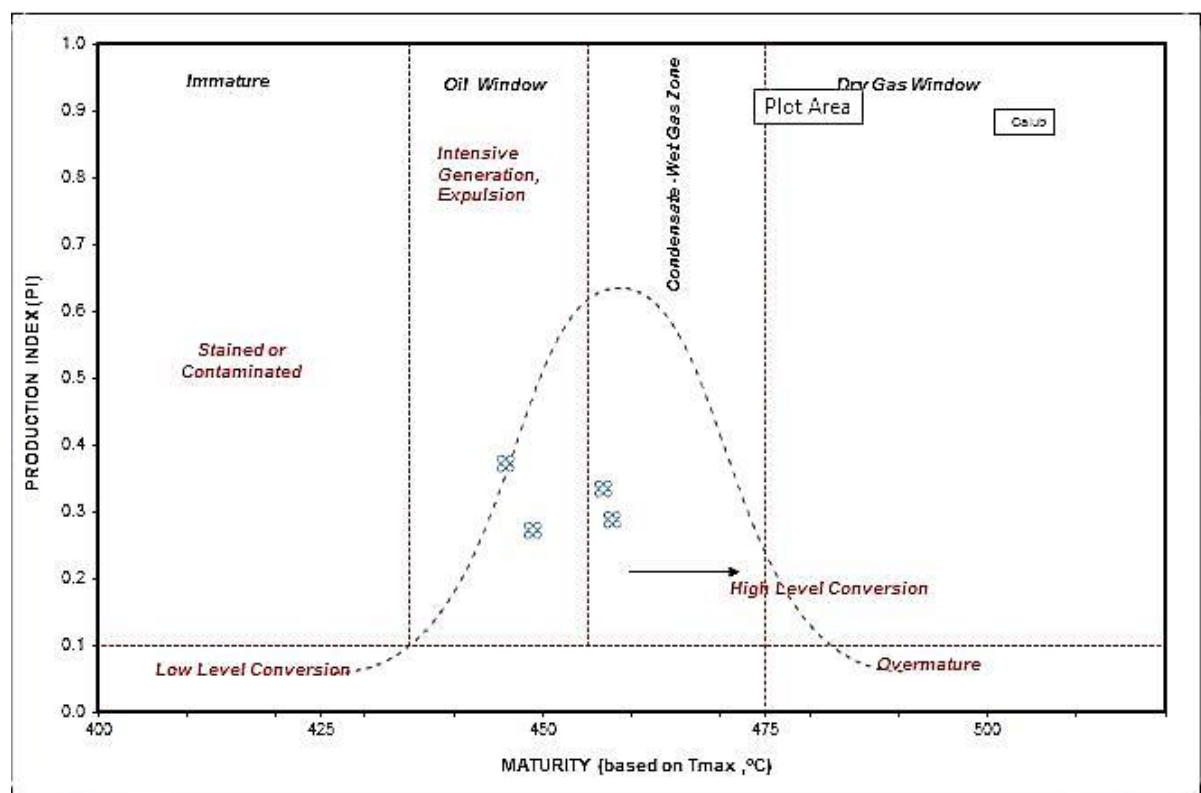

Figure 5.3 Tmax vs. PI cross plot (Ghori, 2002)

5.3   Elemental analysis

Marine shales and mudrocks can be regarded as admixtures of three end-member oxides are: SiO_2 (detrital quartz and/or biogenic silica), Al_2O_3 (clay fraction) and CaO (carbonate

content) (Ross and Bustin, 2009). The ternary plot of the major elements indicates that majority of the shale samples examined are enriched with SiO2 relative to Al2O3 and CaO. The studied shale samples show a high content of SiO_2 (57.4- 59.7 %) with small variations (Table 4.4A). The Al_2O_3 content shows low concentrations (16.9-18.2 %) with small variations (Table 4.4A).
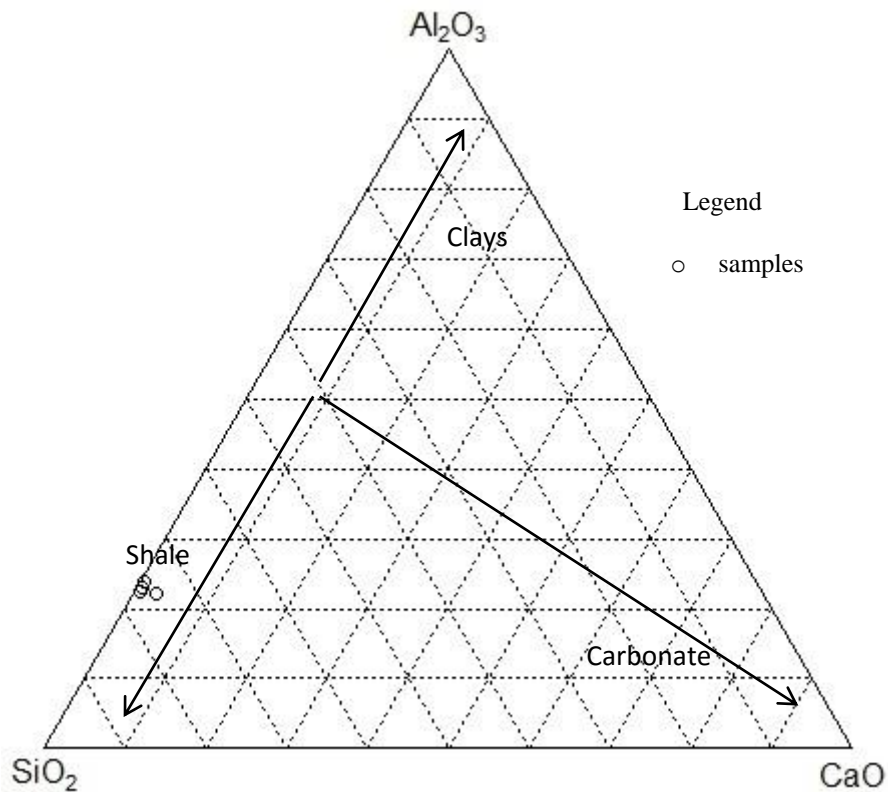

Figure 5.4 Ternary plot showing relative proportions of major oxides (modified after Ross and Bustin, 2009)

5.3.1 Provenance indicator

The geochemical signatures of clastic sediments have been used to find out the provenance characteristics (Cullers, 1995; Madhavaraju and Ramasamy, 2002; Armstrong-Altrin et al., 2004; Nagarajan *et al.*, 2007).

The identification of provenance is based on the elemental ratios, e.g. Zr/Cr, La/Sc, and La/Co ratios can be used to infer the chemical properties of the provenance rocks and, presumably, their signature is preserved in expelled hydrocarbons. They are usually good discriminators between mafic and felsic source rocks because La, Th and Zr are more concentrated in felsic igneous rocks whereas Co, Sc and Cr have higher concentrations in

mafic rocks (Wronkiewicz and Condie, 1987). The ratio of elements provides more reliable information about provenance than the concentration of individual elements. Therefore many authors used elemental ratio to identify the ultimate source material.

Huntsman-Mapila *et al.* (2005) defined the range of elemental ratio that helps to differentiate parent material the clastic rocks are derived from. Accordingly, when Zr/Cr is > 2.0, the source rock originates from a felsic environment and when Zr/Cr is < 1.0, the source rock provenance is from a mafic environment. In the same way Cullers (2000) put the range for La/Sc and La/Co. When La/Sc is 2.5–16.3, the source rock is felsic and when La/Sc is 0.43–0.86 the source rock is mafic. Similarly, when the La/Co value is 1.8–13.8 or 0.14–0.38, the rock is deemed felsic or mafic, respectively.

The studied shale sample has Zr/Cr ranging from 2.36 to 2.65 with an average of 2.47, La/Sc ranging from 3.57 to 4.35 with average 3.92, and La/Co ranging from 2.68 to 3.38 with average 3.04 (Table 5.3). The transitional elements, e.g. Cr and Co; rare earth element (REE) e.g. La and Sc; and High Field Strength Elements (HFSE), e.g., Zr are usually immobile elements concentrated in weathering minerals rather transferred to depositional site as suspended sediments.

Previously the Bokh shale was believed to be derived from nearby basement rock even though it was not supported by geochemical data. It is one of the early clastic sediments that were formed in the Ogaden Basin. The tectonic event dominating at that time was the denudation of the surrounding uplifted basement rock. The samples inorganic geochemical analysis indicates moderate quartz concentration. Therefore this formation is assumed to be unmatured which means the sediment is probably not recycled.

The current study of inorganic geochemical ratios of elements (Zr/Cr, La/Sc, and La/Co) demonstrates that the Bokh shale was sourced from the felsic rock (presumably from nearby granitic rock).

5.3.2   Palaeoenvironment interpretation

Various factors play a role in the preservation of organic matter, notably the oxygen content of the water column and sediment (oxic versus anoxic), primary productivity of new organic matter by plants, water circulation, and sedimentation rate (Demaison and Moore, 1980; Emerson, 1985). Anoxic sediments are typically thinly laminated (distinct alternating layers <2 mm thick) because of the lack of bioturbation by burrowing, deposit-feeding organisms.

Studies by Akinlua et al. (2007) and Galarraga et al. (2008) have shown that source rocks of sapropelic organic matter content usually have higher abundance of V and Ni than those of humic or humic/sapropelic content mainly due to abundant input of porphyrin-precursor chlorophylls to the organic matter derived from algae and bacteria.

Galarraga *et al.* (2008) produced the graphic zonation of V and Ni concentration values which is used as a tool for paleoenvironmental interpretation. When V/Ni >3 and Ni >90ppm suggests the corresponding source rocks were deposited in marine environments under euxinic or very reducing conditions; if V/Ni >3 and Ni <90ppm indicates marine organic material and shale or limestone as source rocks deposited under anoxic conditions; if V/Ni is between1.9 to 3 shows the corresponding source rocks were deposited under dysoxic-oxic conditions with precursor organic matter of mixed origin from continental and, predominantly, marine; and V/Ni <1.9 suggests terrestrial organic material, with prevailing oxic conditions during the deposition of source rocks associated with such organic matter.

The studied samples, Calub 3 shows V/Ni ratio ranging from 2.6 to 2.85 and Calub 5 indicates 3.21 to 3.22 (Table 5.3). This result demonstrates that Calub 3 was sourced from mixed origin of marine and terrestrial organic matter. The lacustrine source rocks exhibits such type of moderate concentrations of Vanadium and Nickel trace element. The V/Ni ratio of Calub 5 is greater than 3 and also the concentration of Ni is higher than 90ppm. Therefore this suggests Calub 5 is derived of marine organic matter. However, the previous study by Worku and Astin (1992) suggested the source rock depositional environment to be large lake based on the structure of the Bokh shale (it is uniform and thinly laminated), the thickness (very thick shale unit), and fauna present and its forms (lack definite marine form). As well as they used lithofacies of shale to interpret the condition under which the source rock was formed. According to lithofacies data the Bokh shale grades upward from clay size material into siltstone and fine sand and this indicates deposition in persistent water body that becomes progressively shallower and more oxygenated over a long time.

The source rock depositional environment determines the proportionality of Vanadium to Nickel. Peters *et al.* (2005) and Killops and Killops (1993 and 2005) used the ratio of vanadium to vanadium and nickel to identify different types of source rock depositional environment. The V/(V+Ni) ratio can be related to redox condition in source rock and low ratio reflects oxic while high ratio (≥0.9) reflects anoxic condition in the depositional

environment. The low V/ (V+Ni) ratio (0.2-0.6) show that the coals are deposited under oxic condition.

The studied samples from both Calub 3 and Calub 5 range from 0.72-0.76 (Table 5.3). This ratio neither suggests the anoxic condition nor an oxic condition but it is an intermediate. Therefore, this result demonstrates that the source rock was deposited under sub-oxic condition. In the previous reports Worku and Astin (1992) used the C/S ratio to test the degree of anoxia and salinity of water body. The ratios they found have a widespread from values appropriate for fresh water lakes to values greatly enriched in sulphur compared to normal marine sediments. Accordingly at the bottom the formation is enriched with sulphur and this demonstrates periods of marked anoxia whereas at the top of the formation the Caron/Sulphur ratio found are typically of those found in fresh water lakes.

Sample name	V/Ni	Zr/Cr	La/Sc	La/Co	V/(V+Ni)
C31	2.825	2.4	4.35	3.38	0.74
C32	2.6	2.65	3.85	3.17	0.72
C51	3.22	2.36	3.57	2.94	0.76
C52	3.21	2.46	3.9	2.68	0.76
Average	2.96	2.47	3.92	3.04	0.745

Table 5-3 ratio of elements. Source: Table 4.4 A and B.

CHAPTER 6

6   CONCLUSION AND RECOMMENDATION

6.1   Conclusion

Because of expensive analysis cost and samples inaccessibility few numbers of samples were analyzed. Depending on the samples analyzed the following conclusions are made.

The results of organic geochemical analysis and organic petrography of the Bokh shale from Calub (2, 3 and 5) have been used to interpret quantity and quality of organic matter as well as to predict the generation potential, and to understand thermal maturity of source rock. In addition inorganic geochemical result has been applied to infer the depositional environment and provenance of source rock.

The overall TOC measure of the analyzed samples of Bokh shale is almost greater than or equal to 0.5wt%. This demonstrates that the source rock contains fair quantities of organic matter. However this source rock seems productive, its generation potential is dependent upon the pyrolysis response because TOC is the overall measure of carbon available in the rock whether it is effective or not.

The pyrolysis result indicates the remaining hydrocarbon or S2 average of 0.27 mgHC/g rock which is less than the minimum required threshold value that is 2.5 mg HC/g rock. S2 is the quantity of hydrocarbon the source rock generates upon further maturation. Therefore the few analyzed samples result shows the remaining hydrocarbon in the rock is not promising or poor in quantity for petroleum generation in the future. This is due to the fact that the Bokh shale has been generating hydrocarbons since a long time and therefore its current generation potential is low.

The average generation potential, i.e. the sum of free hydrocarbon (S1) and remaining hydrocarbon (S2), of the source rock is 0.192 mg HC/g. The hydrocarbon source rock with generation potential less than 2 mgHC/g rock is considered as poor or it is unable to generate petroleum. Hence both S2 and the genetic potential of the studied samples prove the source rock has poor capability of hydrocarbon generation.

The production index of the studied sample ranges from 0.27 to 0.46 mg HC/g rock and it confirms insitu hydrocarbon except the sample with 0.46. This anomaly indicates that the sample was probably contaminated during test well drilling.

The essential rock-eval parameters (TOC vs. S2) and calculated parameters (OI vs. HI) were utilized in order to identify the kerogen types. Both cross-plot of OI versus HI and TOC versus S_2 ensures the Bokh shale is type IV kerogen which means it is dominated by inertinite macerals that generate little or no hydrocarbons during further maturation.

The vitrinite reflectance (Ro) of Calub-5 indicates that shale is in the late mature stage whereas Ro of Calub-3 shows the shale is over-matured (R_o >1.35). Tmax versus PI graph of the shale samples depict Calub-3 is in the wet gas zone and Calub-5 lies in the condensate to wet gas zone except one sample from Calub-2 which is immature. Based on the result obtained, the maturities of the Bokh shale lies between late mature to over-mature.

Inorganic geochemical results (V/Ni ratio) from Calub (3 and 5) suggests the organic matter constituting Bokh shale was sourced from marine, and mix of marine/terrestrial. According to the result from Zr/Cr, La/Sc, and La/Co ratio, the source rock was derived from nearby granitic rock. The V/(V+Ni) ratio indicates that this, the source rock was deposited under sub-oxic condition.

6.2   Recommendation

In this study the whole rock geochemical method of sample analysis is implemented. The kerogen quantity and quality of the Bokh shale were interpreted from pyrolysis data. However, the identification of kerogen quality may face ambiguity upon determination using the cross-plot of OI versus HI, and S2 versus TOC alone. For this reason, the supplementary method has to be adopted for accurate interpretation. The method known so far for kerogen type determination is elemental analysis of atomic H/C and O/C ratio and also this method is non-destructive. Elemental analysis is conducted by kerogen isolation method, but one has to take into consideration that the rock should be good source rock. Therefore in the further studies this method of analysis has to be performed so that kerogen types are better understood.

In this work elemental ratio has been used to the identification of source of the organic matter and source rock depositional environment. This method alone may not appreciably ascertain the source rock depositional environment and precursor of organic matter. Hence, the elemental ratio along with biomarker may assure the source of organic matter and depositional environment of the source rock. Petroleum contains hundreds of compound that can serve as a biomarker. Therefore, the biomarker study which is not conducted in this work has to be implemented in further studies for better understandings of the source rock characteristics.

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Appendices
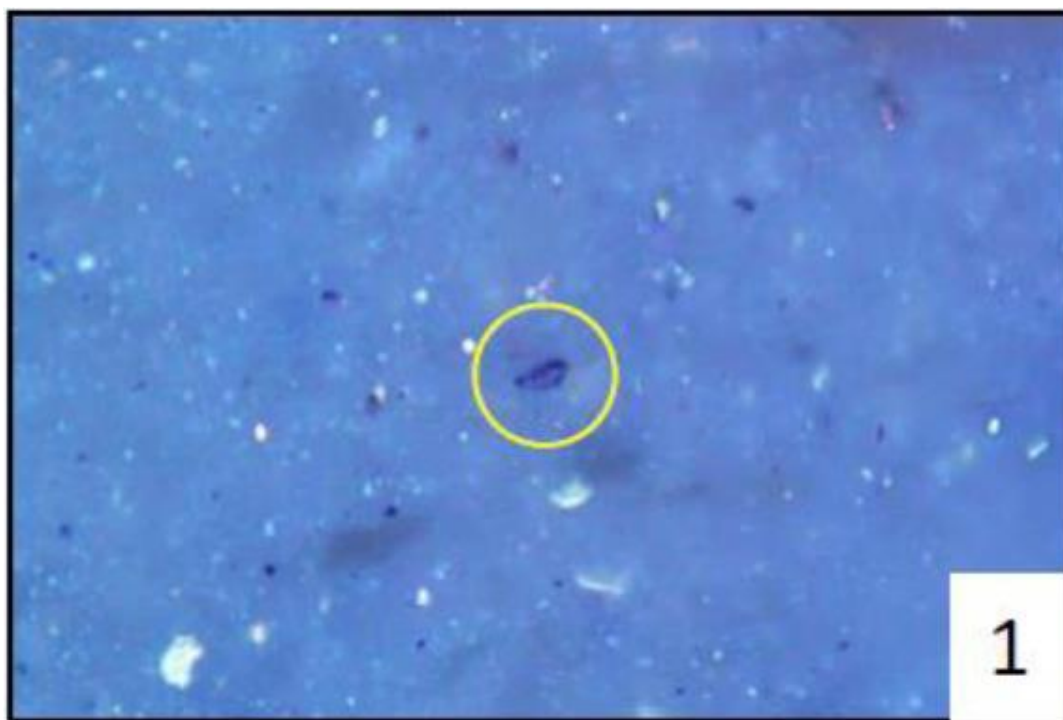
Appendix 1. Organic petrography

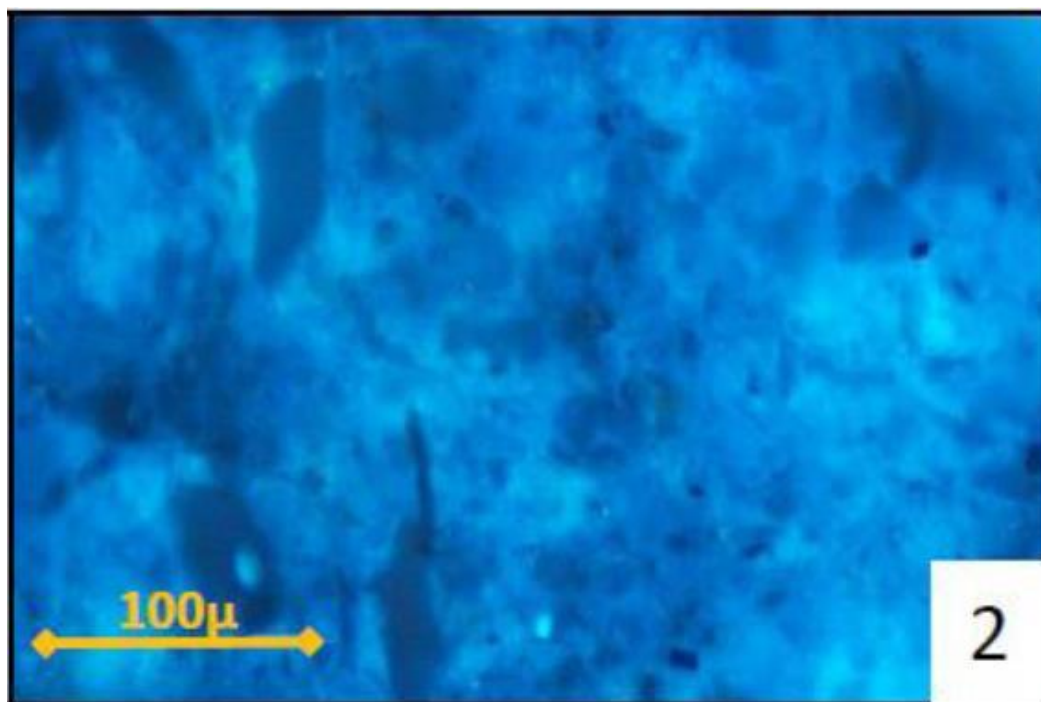Plate 1. Sample C33 white light illustrating in-situ vitrinite particle.

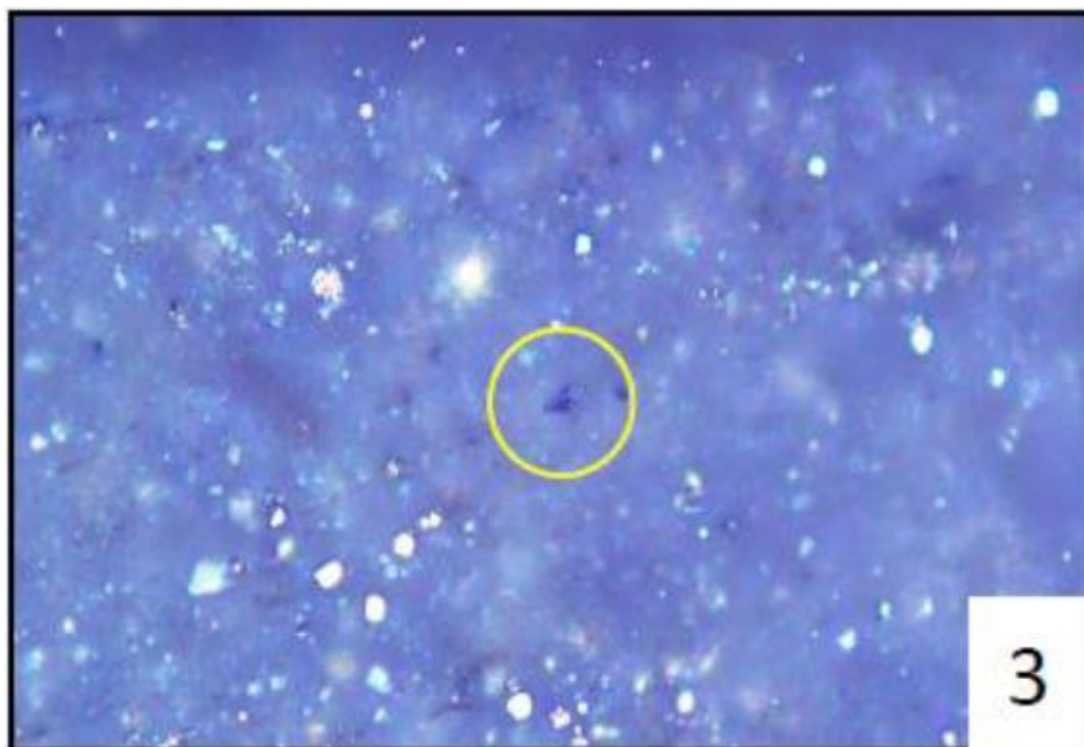Plate 2. Sample C33 UV-light illustrating distinct lack of fluorescence.

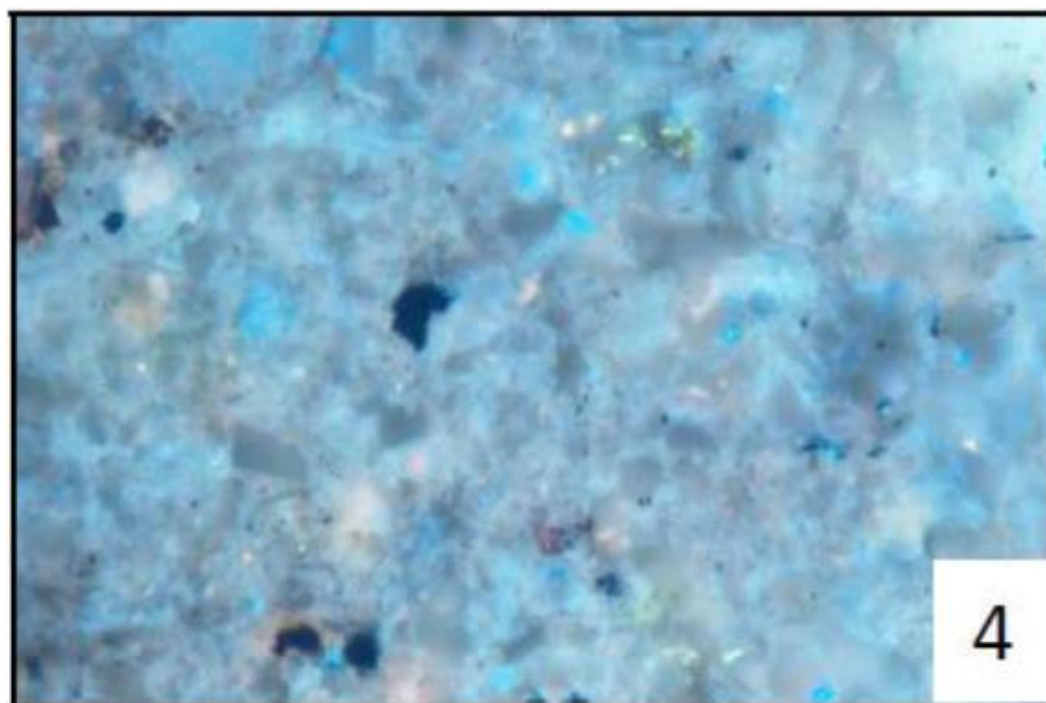Plate 3. Sample C53 white light illustrating in-situ vitrinite particle.

Plate 4. Sample C53 UV-light illustrating very weak fluorescence.