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Research Article

COMPOSITION ANALYSIS AND SOURCE ROCK POTENTIALS OF SOME SHALES FROM THE LOWER SAXONY BASIN, NW GERMANY

Fauziya Ahmad Rufayi[1]., Mohammed Bukar[2]*., Maimuna Halilu[1] and Asabe Yahaya Kuku[2]

[1]Geology Department, Madibbo Adama University Yola, Nigeria
[2]Geology Department, University of Maiduguri, Nigeria

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ABSTRACT

The study involves compositional analysis and source rock potential of samples from Posidonia and Wealden Shales of the Lower Saxony Basin, Germany. Focused ion beam, broad ion beam and scanning electron microscope imaging and point counting were used to study the compositions of these shale samples. Sufficiently large images at high resolution down to nm were studied and statistical representative areas were taken. Qualitative and quantitative descriptions were carried out using Jmicrovision v.1.27 computer software. Total organic carbon (TOC), Rock-eval pyrolysis and vitrinite reflectance were carried out to determine source rock properties (i.e. richness, quality and thermal maturity). In both of the shale units mineral phases and other compositions identified include: dominantly smectite, mixed smectite-illite and near equal proportions of calcite, quartz, mica, cubic and framboidal pyrite and organic matter with subordinate fossil fragments. Samples from both Posidonia and Wealden are kerogen type 1. TOC of 12.59 (Wt %) and 11.8 (Wt %) and vitrinite reflectance of 0.53% Ro and 0.74% Ro were recorded for Posidonia and Wealden samples respectively suggesting adequate organic carbon for petroleum generation but low maturity index.

INTRODUCTION

Shale is one of the most abundant rock found in sedimentary basins. It constitutes the bulk of clastic source rock for petroleum generation. Shale is a fine grained sedimentary rock composed of clay minerals such as (illite, mixed illite-smectite, chlorite) and silt size fragments of other minerals particularly quartz and calcite as well as sulphides (mainly pyrite), amorphous minerals and organics (Blatt *et al*., 1980; Weaver, 1989). The proportions of clay to other minerals are variable, and is characterized by its fissile and brittle nature which allows breakage along the fissile planes. Their potential for petroleum generation and expulsion depends on physico-chemical conditions. Measurements of source rock thermal maturity attempt to describe the progress of the sum of the chemical reactions that convert sedimentary organic matter into oil and gas.

Samples from Posidonia and Wealden shales of the lower Saxony basin (Fig. 1) were investigated for source rock potential and composition. The aim of this research is to determine observable elementary and mineralogical composition and classify kerogen type and maturity index of the samples.

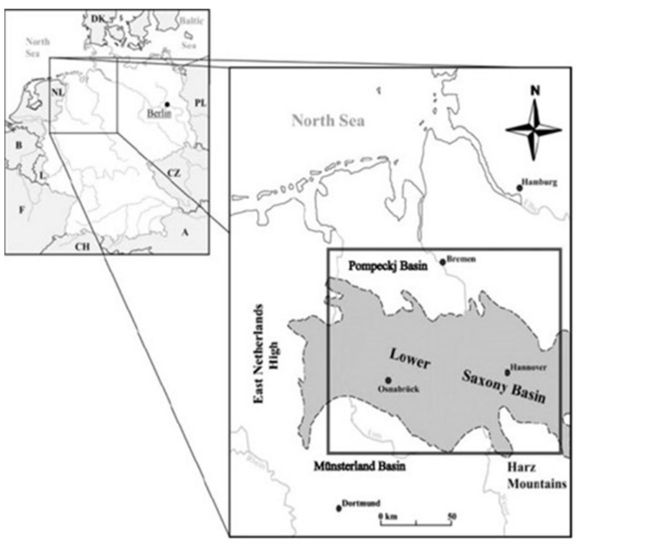

Fig. 1. Location and extent of the lower Saxony basin (after Rippen *et al*., 2013)

Corresponding author:* **Mohammed Bukar
Geology Department, University of Maiduguri, Nigeria

The lower saxony basin

Posidonia shale

Posidonia shale is the second major source rock in the NW Saxony basin, (the major being the Carboniferous coals). This shale was formed in Jurassic Period. It covered an area of up to 500sq km of the Hills syncline area. Posidonia Shale deposits are believed to be anoxic due to the fact that the formation is characterised by deep marine fossils (such as ammonites, sea lilies, schizopores, bivalves, coccolithopores) (Martill, 1993). Posidonia shale in the Hills Syncline could be as thick as 35m with a threefold stratigraphic subdivisions: The upper calcareous shale, middle calcareous shales with bivalve shells and lower marlstone (Fig. 2). These units show minor differences in lithology, though the marlstone unit and shale is distinguishable by the carbonate content within the marlstone (Littke *et al.*, 1991). Posidonia shale is organically rich and lateral variations in its maturity have been related to its deep burial or the effects of Vlotho Massif or professed deep seated igneous intrusion. (Horsfield *et al.,* 2010). Although, a low rank of maturity gradient in the lower Saxony Basin which suggest burial rather than an igneous intrusion (Munoz, 2007).

Wealden Shale

The third main source rock is the Wealden Shale, formed during the lower Cretaceous and is found at the westernmost part of the lower Saxony Basin (Munoz, 2007). The younger Wealden shale well which is known as paper shale of the Cretaceous age occurs only in the western half of the basin. This Formation is brackish to limnic (rare overturn or eruption) in facies. Munoz (2007) documented that the Wealden Shale has total organic carbon up to 8%. Regardless, recent authorities have it that the centre of the Wealden Basin had already been matured and had entered the gas window even before the inversion episode occurred (Munoz, 2007).

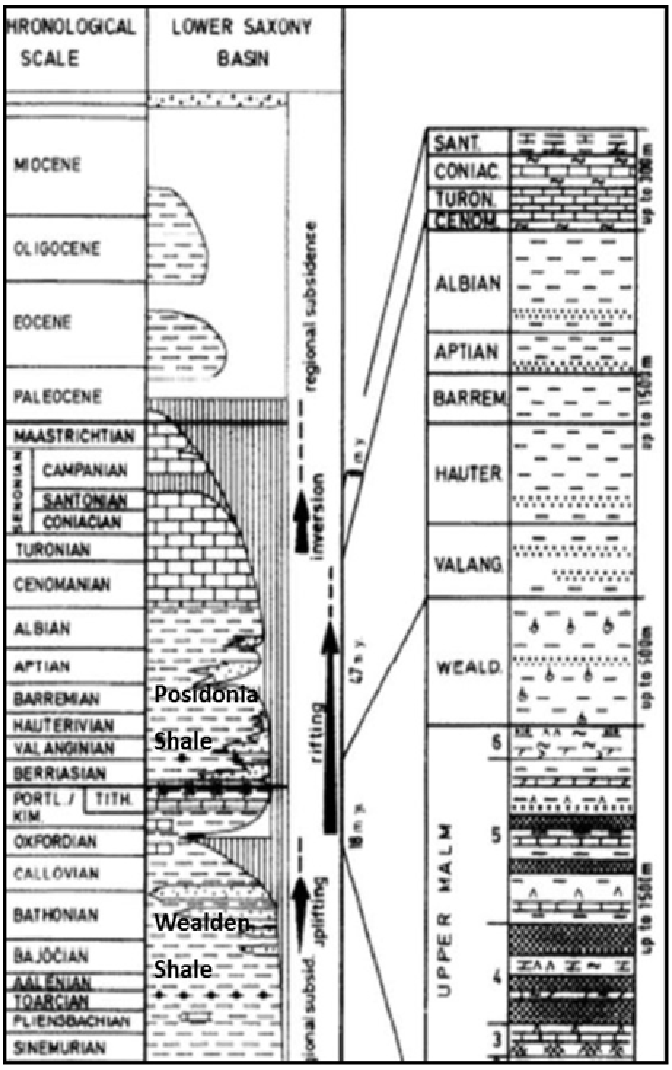

Fig. 2. Tectonostratigraphic Framework of Lower Saxony Basin (modified from Scharzer and Littke, 2007).

METHODOLOGY

The Posidonia shale samples were collected from Schlahe well at the depth of 30.2m. The Wealden samples were collected from Wickensen well at the depth of 923.7m. Both samples are located in the lower Saxony basin of Germany. Samples for geochemical analysis were prepared according to standard organic geochemical (Pratt *et al*., 1992 and Petersen *et al.,* 2000), while organic petrology were determined using the procedures of (Bustin *et al*., 1985 and Taylor *et al.,* 1998). The TOC is calculated from the amount of CO_2 evolved during hydrocarbon generation and also during oxidation at 650°C using Rock-eval 11 apparatus. Maturity of the samples was determined from T_{max} values in degrees Celsius, which is the temperature at the S2 peak maximum. Vitrinite reflectance (Ro) was further employed to determine the maturity of the envisaged source rocks.

Focused ion beam (FIB) and broad ion beam (BIB) together with scan electron microscope (SEM) were used to produces high resolution images to ease qualitative analysis.

Samples were slowly air dried at room temperature at a relative humidity for one week. These samples were weighed before and after drying to estimate the water loss. Polishing of samples mechanically was employed on the samples embedded in a holder to achieve flat cross section, the FIB images were further polished using an acceleration voltage of 30 kV. For BIB cross sections ultrasound cutter was used to cut the samples. These cross sections samples were pre-polished to reduce the roughness and smoothen any flat zone to minimize curtaining effect and produce a better broad ion beam cross section (Desbois *et al*., 2011). This flat cross section allows the surface to be milled to a degree of smoothness which enables the assessment of classifying minerals across the entire surface. Hitachi SU-70 High Resolution Analytical Scanning Electron Microscope, equipped with an Oxford Instrument Energy Dispersive X-ray microanalysis system (INCA Energy 700) was used to produce very high quality images with high magnification in order to investigate the composition qualitatively.

Backscatter electron (BSE) images were obtained using an yttrium-aluminium garnet (YAG) scintillator detector. Microanalysis settings for Energy-dispersive X-ray spectrometry (EDX) collection was conducted at process time 5, collection at 30sec and an acquisition rate of more 2.5K counts per second. Classification of minerals was carried out by point counting method of the Jmicrovision software. Mineral identification on FIB images is based on EDX measurements of BIB images where the FIB-SEM identify minerals and BIB-SEM identify and typify compositions.

RESULTS

Several minerals are near homogeneously distributed in the shale matrix (Plate 1). Seven different mineral phases were identified. This identification was based on the EDX measurement. The seven mineral phases include: clay, quartz, calcite, mica, pyrite, and fossil and organic fragment (Plate 2).

1. Quartz- Medium grey platy grain usually large with smooth edges (Plate 1. 3A, 3B and C).
2. Calcite - Light grey grains show no porosity (Plate 3A and B).
3. Pyrite - Bright to almost white coloured grains which appear in form of octahedral, or as a single large crystal or small aggregates of pyrite framboid (Plate 3C). Pores were identified within the framboidal crystals.
4. Organic matter- Dark to almost black grains which are sometimes confused as pores (Fig.5A and B).
5. Mica - These minerals show elongated grains with parallel alignment on BSE images (Fig.4C, and 5B).
6. Fossil shell- Light grey colour which resembles curvy or bean like structure (Plate 3A and B). Also original fossil morphology been preserved (Plate 3C).
7. Clay matrix- medium grey in colour which seems to be the bulk composition in all the samples studied. Two types of clay minerals were identified smectite and smectite-illite. Clays and quartz have similar grey colour (Plate. 1 and 2A-C).

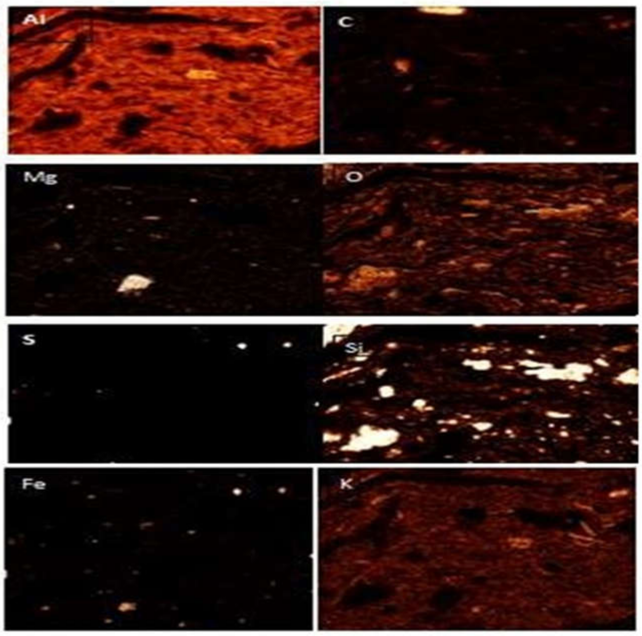

Plate 1. EDXS maps shows elemental chemical composition, (aluminium) Al, Carbon ©, magnesium (Mg), oxygen (O), sulphur (S), silica (Si), iron (Fe) and potassium (K).Matrix forming minerals is mainly Clay (Mg + Fe + O), calcite (Ca + C), quartz (Si +O), smectite (Ca + Na + Al + Mg + Fe), illite (Al + K +Fe + Mg), mica (Si + Al) and pyrite (Fe +S).

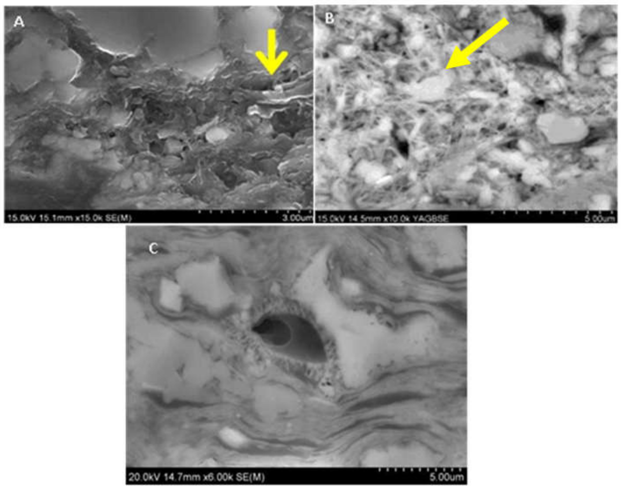

Plate 2. Secondary electron SE images of Posidonia shale showing clay minerals **A.** Smectite (Yellow arrow) **B.** Recrystallized illite-smectite occurs as bridge between grains with boxlike structure sealing pores (Yellow arrow). **C.** SE image of Wealden shale shows illite crossed stalks rimmed at the margin of a pseudo pore.

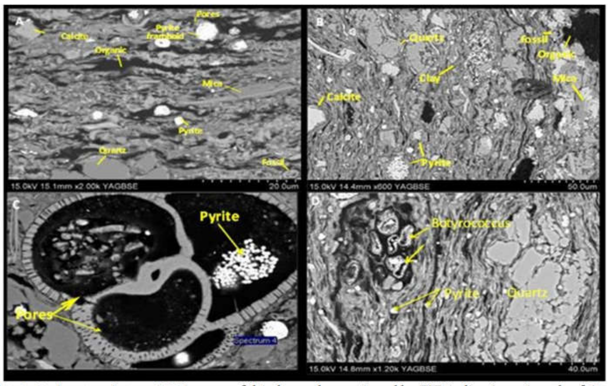

Plate 3. Backscatter electron BSE images of dried samples sectioned by FIB indicating minerals of the samples which comprises mainly of cemented calcite, quartz and pyrite A Posidonia shale and B, Wealden shale. C shows recrystallized coccolithopores preserved in Posidonia shale with pyrite framboids embedded within organic matter matrix of the fossil. D, BSE image of Wealden shale showing botyrococcus fossil preserved within organic matter matrix.

Representative Elementary Area

Mineralogy determination is based on box counting of REA (Kameda *et al,* 2006). Box counting of the REA enabled the classification of 7 different mineral and non-mineral phases (Quartz, calcite, mica, fossil, pyrite, clay and organics; Figs. 3 and 4).

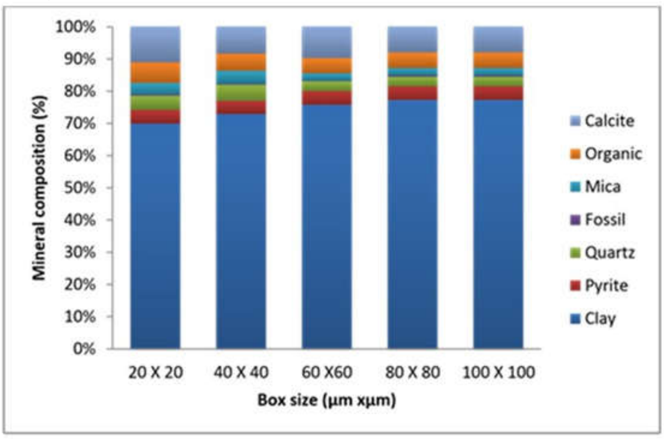

Fig. 3. Point counting results of average mineral composition of Posidonia shale, as a function of square box count showing box size 80x80µm onwards to be representative. Note no specific mineral chosen as starting point.

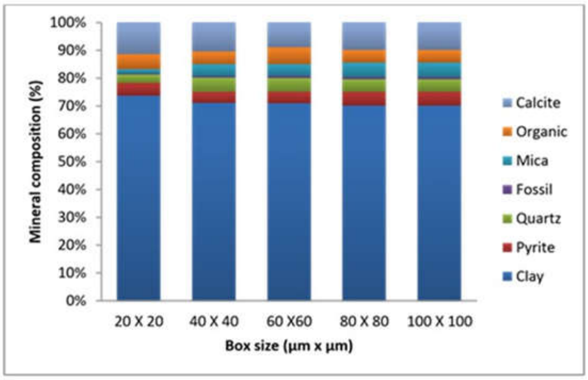

Fig. 4. Point counting results of mineral composition of Wealden, as a function of square box size. Average mineral composition in 2D show consistency at box size 80 x 80 µm onwards, and does not depend on the starting mineral.

Both Posidonia (Fig. 3) and Wealden (Fig. 4) shales show mineral composition as a function of square boxes. At 80 X 80 μm^2 fluctuation in mineral composition becomes negligible. Therefore, the box size at the scale on the BIB cross section is said to be representative. This box counting methods was carried out with no specific starting point.

Hydrocarbon Potential of the Study Area

This study used T_{max} vs. hydrogen index (HI) (Fig. 5) to illustrate the kerogen type of both Posidonia and Wealden samples. Both samples are kerogen type I which indicates that samples were likely to have been deposited under anoxic conditions. Type I kerogen mainly consist of algal materials such as alginite, coccolithopores, cynobacteria etc. Table 1 also shows the maturities of samples are 0.58 % RO and 0.74 % Ro for Posidonia and Wealden respectively.

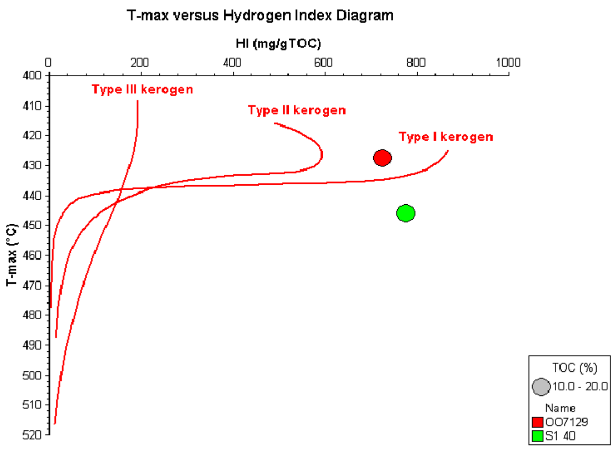

Fig 5 Shows the kerogen types of Posidonia (Red) and Wealden (Green).

Table 1 Properties of Posidonia and Wealden shale.

Properties	Posidonia	Wealden
Age	Toarcian	Barriasian
Well	Wickensen 007129	Schlahe S140
Depth (m)	30.2	923.7
Fossil	Coccolithopores	Botryococcus
TOC (Wt %)	12.59	11.8
HI (g/mg	725.55	775
Tmax (oC)	427	446
Vitrinite (% Ro)	0.53	0.74
S1(mg/g)	4.98	0.93
S2(mg/g)	91.31	91.48
PP (%)	92.41	95.89

DISCUSSION

Composition of Shales

The high resolution technique used enabled the identification of six different minerals (clay, calcite, quartz, mica, pyrite and organic). Bulk mineral composition of both Posidonia and Wealden are represented in a bar chart (Fig. 3 and 4). Both studied samples reveal clay mineral having the bulk composition. Representative elementary areas illustrate point counting results of average mineral composition of Posidonia and Wealden shales, as a function of square box count showing box size 80x80 μm^2 onwards to be representative.

Fossils observed in this study samples reveals calcified fossil forms as well as well-reserved structures. Coccolithopores (Plate 3C) in Posidonia shale show imprint of fossil in clay matrix which is calcified with nanoscale pores visible whereas botryococcus (Plate 3D) in its original biological form preserved in organic matter matrix of Wealden shale. Presence of these fossils could have been controlled by high stand system track in the environment of deposition when there was a fair weather condition for diversity.

Clay Minerals

Posidonia and Wealden samples show the presence of smectite and smectite-illite mineral and fibrous bridge illite crosscutting each other. In Wealden shale presence of these clay minerals at 923.7m and 30.2m depth suggest that they are in illite-smectite (I/S) mixed layer. Smectite transformation to illite has great effect on physical properties (Aplin and Macquaker, 2011) since clay rich minerals are deposited as flocs (Bennett *et al.*, 1991). Expanding layers of illite-smectite decreases as a function of burial diagenesis and geothermal alteration (Velde, 1985). Smectite dissolution increases the silica content of illite through illitization process (Boles and Franks, 1979). Walderhaug, (1986) also considered precipitation of illite and pore destruction as the principle factor contributing diagenetic porosity loss. This is due to the time, temperature and chemical variation which simultaneously affect the illite-smectite reaction series (Velde and Vasseur, 1992). The variation in depth of smectite can vary from basin to basin. Therefore, this idea cannot be generalised for all basins. The figure below (Fig. 6) illustrates generalised diagenetic sequence of clay minerals as a function of depth and temperature.

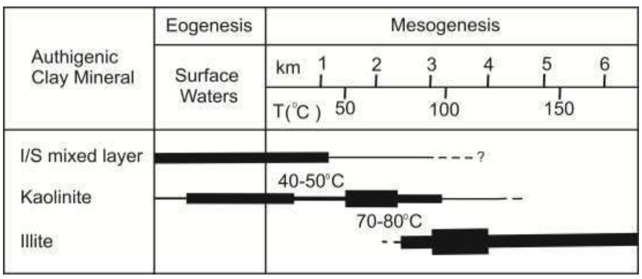

Fig. 6. Shows diagenetic sequence of clay minerals. The thickness of each line approximates the abundance of each mineral over a particular burial/temperature range. (Burley and Macquaker, 1992).

Hydrocarbon Potential

The geochemical pyrolysis parameter Table 1 shows properties of study samples. Rock Eval T_{max} is the maximum temperature at which hydrocarbon is converted to kerogen (Espitalie *et al.*, 1977). Fig. 8 illustrates that both Posidonia and Wealden shale are kerogen type I. Kerogen type I is derived from reworking of lipid-rich algal debris by bacteria (Peter and Moldowan 1993). Classifying Posidonia shale as type I kerogen contradicts literature that Posidonia shale is type II kerogen (Baskin, 1997). This could probably be because samples were taken from the different sections of the basin. TOC of 12.59 (Wt %) and 11.8 (Wt %) are adequate for petroleum generation. The low maturity of 0.53%Ro and 0.74%Ro of Posidonia and Wealden samples respectively, suggests that organic matter (kerogen) was not converted to hydrocarbon. This clearly indicates that samples are at early and mid- maturity zone respectively.

CONCLUSION

The studied samples exhibit same mineralogy with varying amount of quartz, calcite, fossil shell, pyrite, mica and clay minerals even though were taken from different wells and at different depths. Clay mineral is the bulk composition 70% and 74% for Posidonia and Wealden samples respectively. These comprise smectite, illite, mixed illite-smectite with no chlorite. Posidonia shale is composed of imprint of recrystallized coccolithopores fossil remain whereas Wealden shale is composed of original biological form of botryococcus fossil. Rock Eval and T_{max} value plotted against Hydrogen index (HI) revealed that both samples are kerogen type I and of low maturity.

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