Microscopy of Pore Types in the Belata Black Shales: Implications of Gas Storage*

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Abstract

Rock properties like permeability, toughness and durability can be examined through pore structures. Randomly selected 22 black shale samples from the Belata Formation were studied to identify their morphology and pore structures with the use of Scanning Electron Microscopy (SEM). The analysis identified four main types of microstructures (pores) in nanoscale. These are mainly the inter-particle microstructures, organic microstructures, micro-fractures and intra-particle structures. The inter-particle microstructures occur between clay and quartz particles and the organic microstructures are large enough to accommodate gas molecules.

Keywords: Pore Structure, Shale Gas, Scanning Electron Microscope, Belata Formation, Black Shale

1 Introduction

Migration, accumulation and production process of shale gas is affected greatly by the pore structure (Tang et al. 2021). Techniques like the scanning electron microscopy, (SEM), fluid injection which includes mercury intrusion porosimetry, (MIP) and N_2/CO_2 gas adhesion can be useful in characterising of pore structures in tight formations such as shales (Clarkson *et al.,* 2013). SEM technique is applied on shales of fine-grained texture. The technique produces clear images of pore structures for rock characterisation. These combined with statistical methods, can be used to quantitatively characterise different pores (Jochum *et al.,* 1995). The differences in pore type can affect the geochemical characteristics of the rock (Tang et al. 2021). These geochemical characteristics are relevant to the stability of the wellbore and hydro-fracturing (Slatt and Abousleimann, 2011).

The study of the heterogeneity of the composition of organic matter, pores present in the organic matter, the size and types are all easily identified under the scanning electron microscope (SEM) images. It is needful to understand the contribution of organic pores amongst other parameters to the entire pore system in black shales (Tang et al. 2021). This facilitates a better understanding for the evaluation of all black shale source rocks or reservoirs. Such influences on the pore structure will in turn control petrophysical properties which includes permeability, porosity, formation of resistivity factor and irreducible fluid saturation.

1.1 Geologic Setting

Peninsular Malaysia is located within the Sibumasu Terrane region. It is geographically sectioned into three main regions by trending belts which runs from the north and the south based on stratigraphic differences, structural magmatism and geological evolution of Belata Formation (Fig. 1). The Belata Formation forms part of the Western Belt of Peninsular Malaysia. The formation ages from carboniferous and ends in the Permian age. It is stratigraphically correlated with the Kenny Hill Formation (Gan 1992). It is situated within boarders between the southern part of Perak and Selangor, which is locally known as the Tajung Malim town. It lies between the Terolak formation which is to the north and the Kerak formation which is to the south. (Fig. 2). The formation is named after Bukit Belata, which used to exist as a forest reserve. The areal extent of the formation covers approximately 259km^2. The lithologies present are the argillaceous facies of phyllite and shale seen at the base (which is in the carboniferous age) and a principal arenaceous facies which are the metaquartzite and metasandstone up the formation (Permian age). There are chert intercalations in the lower part of the formation (Peng et al. 2004) (Fig. 3).

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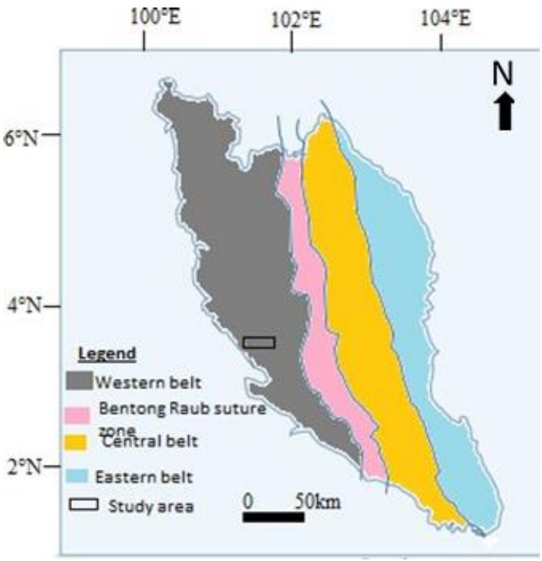

Fig. 1 The Study Area (Metcalfe, 2013)

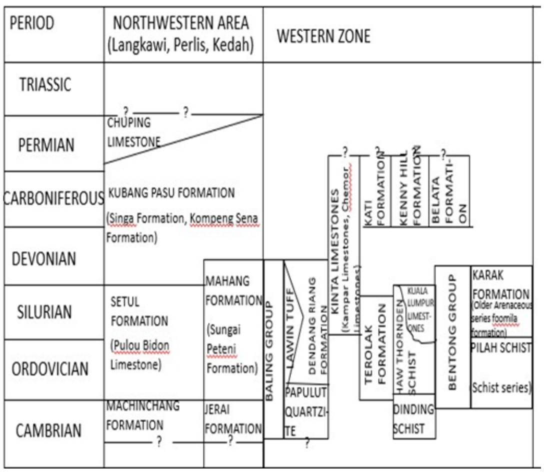

Fig. 2 Stratigraphy of Belata Formation within the Western Zone (modified after Foo, 1983)

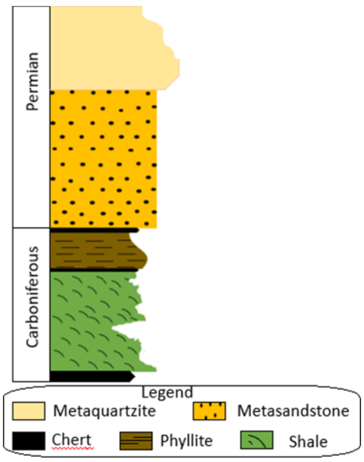

Fig. 3 Stratigraphic Section of the Belata Formation

2 Resources and Methods Used

2.1 Methods Used

Twenty two samples of black shale from the Belata Formation were used in this study to classify and report on the varieties of pores and to interpret their controls on their potential storage of hydrocarbons. This will be carried out by employing the scanning electron microscope (SEM) to identify the pore types. The porosity of a rock refers to the percentage of volume of the rock that is occupied by voids that exists between the rock grains. The inter-particle pores in this study refers to those pores between two different grains of sediments or crystals whilst the intra-particle pores or cracks are those pores found between one solid crystal or sediment grain that may have been broken. Organic pores refer to pores with their porosity in organic matter ranges from 0 to a little above 5% whilst black shale increases in maturity. Organic pores actually are small in diameter and in the large organic particles, they have little connection to the mineral matrix. Finally, micro fractures are fractures caused by tectonic activities that may have affected the rock.

2.2 Petrographic Analysis

Twenty two fresh black shale samples were gathered through a two week intensive field work which covered the entire stretch of six outcrops within the Belata formation. These outcrops were exposed by road and railway cuts due to industrialisation as shown (Fig. 4). Before the samples were taken through the microscopic analysis they were washed in a sieve with water to remove any unwanted or weathered parts. The samples were after that analysed using the scanning electron microscope (SEM) technique to understand the pore structures in the black shales. The scanning electron microscope is mostly employed to replace the traditional petrographic thin sections as it gives way for fine grained sediments to be considered and analysed at higher resolutions. The fresh samples were slashed and polished into thin blocks and sprayed with gold for compositional and morphological identification of minerals with the application of the scanning electron microscope equipped with the energy dispersive x-ray spectroscopy (SED\EDX). Digital images and variable pressure field emission scanning electron microscope was employed.

The electron beam interacts with the atomic structure of the samples and this further produces signals with information on the surface and topography of the sample, composition and other properties. Identification of mineral structures were conducted based on Welton (2003) and interpretation of morphological textures carried out based on Helland *et. al.*, (1997), Abu-Zeid *et. al.*, (2001).

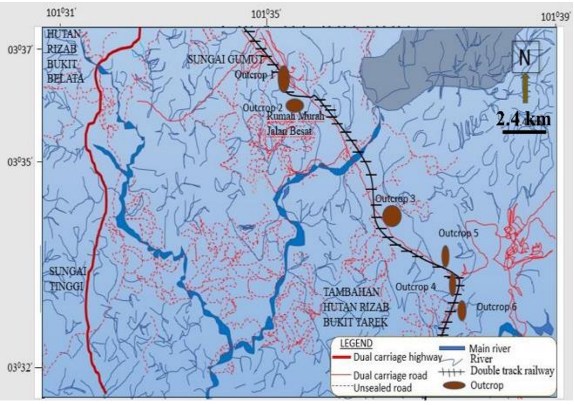

Fig. 4 Map of the Study Area

3 Results and Discussion

3.1 Mineralogical Composition of the Black Shales

Quartz, muscovite, feldspar, pyrite, calcite, kaolinite and chlorite were identified using the scanning electron microscope. According to Fig.5, there is a visible presence of organic matter mixed homogeneously with the clay and other minerals in the black shales. The quantification of these minerals in a pictorial view is summarised by the work of Boateng *et al.*, (2018) together with the mineral's properties. Black shales of the Belata Formation are mainly composed of silt-sized quartz with grain sizes varying between very fine –grained to silty The quantitative measurement of mineral sizes has been worked on by Boateng *et al.*, (2018). The second dominant minerals of the black shale are the clay content (chlorite and kaolinite). Locally, the percentage of quartz, clay and feldspar between the outcrops vary. Hematite occur as interstitial minerals within some pores. Samples from outcrops 1-3 show abundant quartz with a decrease in clays; Samples from outcrop 4-6 show a decrease in quartz minerals but increase in the clays comparably (Table 1). On the other hand, the calcite and pyrite concentrations were found in traces. From the SEM results, kaolinite was seen as pseudohexagonal plates or books (Fig. 8). Kaolinites present in the samples which has been as confirmed by further mineralogical analysis backed by X-ray diffraction reported by. Fig. 6 to 9 shows quartz, potassium feldspar, chlorite, kaolinite and pyrite which was also confirmed in the work of Boateng *et al.*,(2018)

Table 1 Modal percentage of minerals in the Belata Formation

Mineral	Modal Percent Range
Quartz	56- 80
Potassium Feldspar	15 -25
Kaolinite	2-5
Chlorite	2 -15
Pyrite	>1
Muscovite	2-5

3.2 Pore Texture of the Belata Black Shales

Fig. 5 to 9 shows the morphology features of the black shales and also demonstrates the chemical composition which includes quartz, potassium feldspar, chlorite, kaolinite and pyrite of the samples and the macroscopic characteristics of the pore structure. The observed pore types in the black shales from Fig. 5 to 9 are the interparticle pores, organic pores, intraparticle pores and micro fractures. Microscopic characteristics of organic pores are demonstrated in Fig. 5. These pore structure occurs between the organic matter composition which is homogeneously mixed with other silt to clay sized minerals grains of the black shales. Fig. 6 shows a silt size quartz grain with micro fractures. Fig. 7 is a photomicrograph of intraparticle pore sizes between broken grains of potassium feldspar. Fig 8 shows the images of chlorite and booklets of the kaolinite with an interparticle pore between separate mineral grains. Fig. 9 shows a SEM photomicrograph of pyrite surrounded by interparticle pores.

Shale has pore structures that greatly impact shale gas storage capacity (Slatt and O'Brien, 2011). The microscopic features of pores largely determine how shale gas reservoirs function. (Ambrose *et al.*, 2010). The various pores detected in the selected samples from the six different outcrops are convincingly large enough as shown in Table 2, to store producible hydrocarbons if present in an unconventional system. From the recent works done by researchers in the last few years, researchers have found different pore types in different shales by the use of the SEM method as done in this study. Slatt and O'Brien, (2011) acknowledged six varieties of pores. The first of them is the organo-porosity formed during burial and maturation. The second is the inter-particle pores formed as a result of flocculation. The third is the intra-particle pores and this found in mineral grains. The fourth is intra-particle pores from organisms and micro fractures. The fifth is micro channels inside the shale matrix and finely micro fractures as a result of tectonic activities. The works of Shoieb *et al.*, (2017) and Loucks *et. al.*, (2012) show three major pore types classified by organic matter pores, inter-particle pores and intra-particle pores and these are also seen in this research. Comparatively, SEM gives a better picture about the actual size of pores distributed in the shale samples than N_2 adsorption (Table 2).

Table 2 Pore Type and their Measurement

Pore type	Measurement (μm)
Organic pores	2-10
Micro-fractures	2
Intraparticle pores	4-10
Interparticle pores	2-6

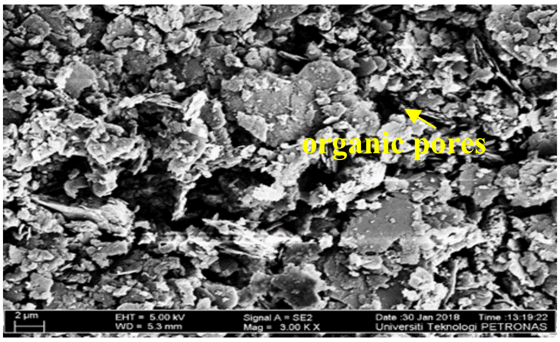

Fig. 5 Back Scattered Photomicrograph of a Homogenous Mixture of all Black Shale Minerals

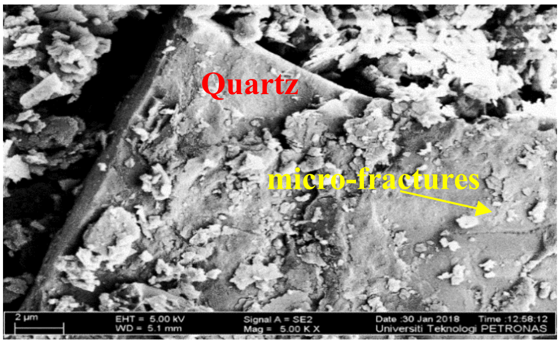

Fig. 6 Photomicrograph of Quartz in the Belata Black Shales Showing the Pore Spaces Created by Microfracturing

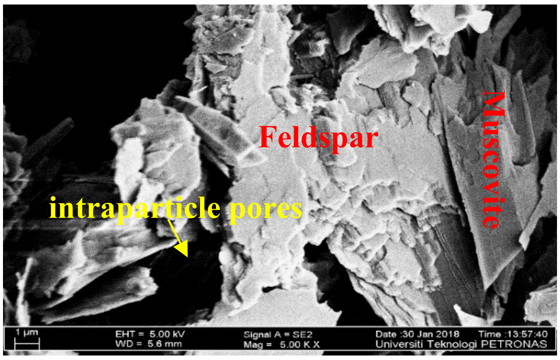

Fig. 7 Photomicrograph of Muscovite and Potassium Feldspars in Shales Showing the Intraparticle Pore Spaces

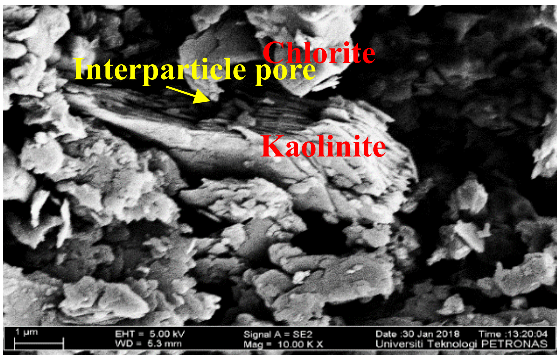

Fig. 8 Photomicrograph of Clays in Shales Showing the Interparticle Pore Spaces

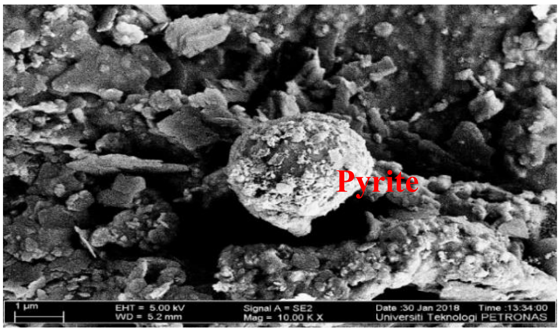

Fig. 9 Photomicrograph of Pyrite in Shale

4 Conclusions and Recommendations

The black shales of the Belata Formation in Western Peninsular Malaysia shows complexity of their pore network structure. These are inter-particle microstructures, organic microstructures, intra-particle microstructure and micro fractures. The pores are well developed and large enough at nanoscale to act as storage for hydrocarbons.

Further effective pore analysis are recommended for study after the gas have been confirmed viable and the black shales identified as source rocks to unconventional resources A deeper knowledge about these pore types will contribute greatly to porosity in the samples and thereby enhance storage of these resources.

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