

## Mineralogical Description and Pore Size Description Characterization of Shale Gas Core Samples, Malaysia

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**ABSTRACT:** The most important properties in shale gas plays are the mineralogical content and its pore size distribution. For many years, shale formations were viewed as a hydrocarbon cap rock. Due to this traditional point of view, up to date only geochemical analysis has been routinely performed on the shale layers. However, for sweet spot mapping of shale gas plays, it is very important to know about petrophysical properties. Shale gas, which is mostly methane can be found in any sedimentary basin, but special methods of extraction is needed. Shale gas can be more expensive than conventional gas to extract. The main outcome of this study was to identify the mineralogy and pore size distribution of samples collected from Kedah and Batu Gajah, Malaysia. Both samples were poorly understood and described on the open source thus investigating these properties on the samples were a good start in differentiating between sandstone and shale rocks. The first section of this paper discusses the different setting of shale in varying environments. For investigating the mineral properties which plays a significant role in controlling shale properties, the experiment x-ray diffraction has been carried out. By carrying this compositional analysis many different type of elements were identified. This was a very important step to identify the right elements using search and match procedures. The second section of this paper discusses the pore size distribution. Pore size distribution in shale gas ranges can be measured using Brunauer-Emmett-Teller

**KEYWORDS:** shale gas, mineralogy, pore size, organic content, Malaysian basin

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### I. INTRODUCTION

The term shale gas refers to produced gas from fine-grained sedimentary rocks which are organically rich in shale. Since conventional gas is produced from granular, porous and permeable sandstone formations, shale gas is considered to be an unconventional gas resource (Beckwith, 2011). In this particular case, the term an unconventional refers to the type of rock which the gasses are produced from, thus the term shale gas is generated. This classical low permeability rock has a sealing lithology of trapping mechanism, where it avoids hydrocarbons to escape vertically or into other formations.

Moving on, organic-rich shale can be divided into three main categories which are type 1 kerogen, type 2 kerogen and type 3 kerogen (Liu et al., 2017). Lacustrine shale is normally associated with type 1 kerogen because of algal source rich in lipids and also organic matter being associated with it. Type 2 kerogen is very much affiliated with the mixture of phytoplankton, membranous plant debris and bacterial microorganisms in marine sediments. Whereas, marine- terrigenous coal bed carbonaceous shale is closely associated with type 3 kerogen because of its high plant debris and more commonly found at Delta tops.

Furthermore, due to cost prohibitive to drill large quantities to achieve economically recoverable gasses, shale gas wells are currently only viable onshore. Generally, these wells are also drilled horizontally to maximize exposure to the reservoir but in some rare cases where the shale interval is thicker than usual, vertical drilling will be carried out. This technically recoverable shale gasses have a very complex petrophysical property which includes mineralogy, porosity and permeability. The main aspect that plays an important role to investigate the performance of fracture treatment in shales is mineralogy. This is because, in terms of mineralogy, fracking treatments are normally more favorable in brittle mineral where the clay content is much higher. The shale minerals tend to deform while hydraulic fracturing rather than shattering.

Based on **Figure 1** shale gas is forecasted to provide a great contribution to domestic gas production. There are also some environmental challenges when reservoir simulation and big scale hydraulic fracturing is done. During hydraulic fracturing process, the potential of water aquifers to be contaminated is very high. This is due to fracturing fluids where it includes using sophisticated and robust techniques to produce a control treatment reflecting in situ reservoir conditions. Moreover, in more arid and deserted areas, where water resource management is more critical, freshwater is only required for a short period of time for a complete treatment of gas shale wells (Alyousuf, Algharbi, Algeer, & Samsudin, 2011).

In addition to that, geomechanical properties of shale gasses which are influenced by clay content play a very important role to determine the source rock geology. In order to identify sweet spots of shale gasses, the early exploration phase of facies association basin history analysis and the sequence stratigraphy must be conducted. The estimated ultimate recovery per well should be the central aspect when evaluating the economic viability of these type of reservoirs.

Shale gas, which is mostly methane can be found in any sedimentary basin-in other words, virtually anywhere in the world. Shale gas is extracted from a geological structure known as the source rock in which natural gas is generated. The gas formed when organic-rich sediments were deposited on the bottom of the oceans and lakes, and then gradually covered over by additional sediment layers. A rock which can be breakable into thin structures and which comprises of mainly sedimentary rocks are where shale gas can be normally formed. Shale has low matrix permeability, to carry out carry out commercial production in these types of reservoirs, fracturing procedure will have to be carried out (Alyousuf et al., 2011). The free gas resides in fractures and pores in shale and is easier to produce relative to the gas adsorbed (Wang & Reed, 2009). That is why the initial rates of production are higher in shale. They decline rapidly to a low steady rate within about one year as adsorbed gas is slowly released from the shale. Mineralogical content in shale gas basins diverges extensively.

## II. MINERALOGICAL DESCRIPTION OF SHALE GAS

**Figure 2** demonstrates the plot established on quartz, total clay and carbonate content based on Barnett and Eagle ford shale samples. The compositional differences span nearly the whole compositional field of this design. As can be seen from the graph, almost more than 50% contain quartz or carbonate. This means that if a good stimulation is carried out these shale gas region would have a better response. Brittleness of the rock, in this case, can be considered as a good one. The difference in the mineralogical content of shale gas plays can be measured as small as to the cm scale. The main controlling factor is shale gas plays are the mineralogy content. Methane adsorption will normally occur on the surface of clay minerals; thus the presence of clay would be a very a vital factor. No clay minerals such as quartz are very vital to determine the brittleness of the rock. The term brittleness mainly refers to tendency of the rock to fracture and can be equated using the formula given below. The most common technique to measure the mineralogy content of a rock is by using the X-Ray Diffraction.

**Figure 3** shows of tinny photomicrograph from a mudstone demonstrating mm-to-cm scale tinny beds of mud rich and carbonate-rich. The lithology that tends to generally exceed and also can be found in mudstone are the lithology heterogeneity. Majority of the number of sedimentary rocks are made up of shale. Shale basically consists clay size minerals and can be referred to as mud rocks (El Nady & Hammad, 2015). This sedimentary rock contains at least 50% of clay and silt-sized particles. For further inspection, clay is compounded by clay minerals such as illite and kaolinite, and non-clay minerals such as silica and carbonate. Furthermore, heavy mineral grains such as iron oxide and organic materials are present in shale rocks. Thin layers and thin beds of limestone make up the rock as a whole. To inspect the quality of shale reservoirs to be a potential candidate for gas production is by examining its organic content, depth and pressure, permeability and thermal maturity. Shale has very low permeability and thus to start production from a shale gas reservoir, advanced methods of stimulation would be highly justified.

This mineralogical tool has a very complicated theory which is mainly based on neutron-induced concept. It basically captures gamma ray spectroscopy to measure yield in an elemental perspective. This tool uses the help of americium-beryllium which is a chemical source. This chemical source emits neutrons to the surrounding environment. The neutron energy decreases with time and interactions. The data which are analyzed are actually the emitted gamma ray. The acquired neutron induced, capture gamma ray undergo special fitting to give the relative elemental yields of eight different elements. Among the most common elements are the magnesium, potassium and calcium. Moving on to give elemental weight fractions oxides closure model is applied. From the elemental weight fractions, mineral volumes are interpreted. These weight fractions are normally coupled with neutron porosity, bulk density and volumetric adsorption data. The main approach that is used in mineralogical interpretation is by using probabilistic error-minimization approach. All these data can actually use to solve mineralogy and porosities value.

A very wide range of minerals is present in shale, which can be determined using the X-Ray Diffraction. Clays have a definite structure comprising alternate layers of silica and tetrahedral alumina with a confident layer of exchangeable cations. The main factors that would affect the type of clay in the minerals is the rock deposition type and climate. Depositional environment does not alter the framework of clay, but there is a considerable amount of exchangeable cation population (Dargahi, Rezaee, & Pejicic, 2013). The most abundant in a clay mineral shale is illite. The mineral illite mainly consists of a repetition of a good amount of tetrahedron-octahedron-tetrahedron(TOT) layers. Some researchers also call this mineral as the clay-sized alumino-silicate mineral which does not expand. It is also the main mineral found in deeply buried shales layers and thus it can be derived from pre-existing shale minerals. This mineral is significantly associated with chlorites and be classified as 1M and 2M. The chemical formula which is given to illite is  $(K, Ca)Al_3Si_3O_{10}(OH)_2$ . With increasing pressure and temperature where the mineral is deposited, the end product of illite is muscovite (Mishra et al., 2018). There are many varieties of illite. A very significant and special variety is the iron-rich mineral, which can be known as glauconite. It seems to be exclusively marine and forms during slow sedimentation.

Kaolinite forms in soils developed under abundant rainfall, good draining and acid waters. During subtropical and tropical weathering, these type of kaolinite characteristics can be found. In the oldest and ancient basins, these kaolinite minerals can be found near the shore. This shows an important indicator and pointer of its special geological setting. The chemical formula which is normally associated with this mineral is  $Al_2Si_2O_5(OH)_4$ . Basically, these mineral has a layered silicate mineral. The is linked with one tetrahedral sheet of alumina and one tetrahedral sheet linked through oxygen (Dargahi et al., 2013). Kaolinite is a soft and most often white mineral produced by the chemical weathering of aluminium silicate minerals like feldspar. The main colour of kaolinite is a red and orange colour which mainly comes from iron oxide. The normal percentage of quartz in an overall shale is approximately 20%-30%. Quartz also can be found on earth surface very abundantly. Some of the components such as sedimentary rocks and metamorphic rocks have a significant amount of quartz in them. The main system that quartz belongs to are the trigonal crystal system. The main idea behind the system occurs at the molecular level with six sharp pointy ends at the ends of the prism.

### III. PORE SIZE DISTRIBUTION OF SHALE GAS

Determining the characterization of pore size distribution (PSD) and surface area of shale is a very demanding task due to their very small pore structure (Zolfaghari & Dehghanpour, 2015). The presence of clay minerals also makes it very hard for experiments to be carried out successfully. PSD mainly controls the main petrophysical properties and it can be used to characterize rocks from different reservoirs (Song et al., 2018). A relative study shows indicates that different methods give different results of PSD. However, based on all calculated and determined PSD, most of the values obtained are smaller than 10nm.

There are two main methods to determine the PSD in shale gas samples. Which are by using mercury injection capillary pressure (MICP) and by Brunauer-Emmett-Teller (BET) analysis to achieve the adsorption and desorption isotherms. Before any of the two experiments can be carried out, the samples must be crushed. The first reason is that the low porosity of the sample may result in an incomplete penetration. Thus by crushing the samples, more area can be assessed by the gas and the accuracy of measurement will improve. Another good reason to crush the sample is to reduce the artefacts and also the microfractures.

Rock Physics model provides some basic important links between macroscopic and microscopic rock properties and basics for predicting rock properties from geophysical data. Some important are the properties of shale gas and organic matter. This is because not only it has unique velocity, density and resistivity, but also it is a good indicator of gas production potential (Sone & Zoback, 2011). **Figure.4** demonstrates the complexity of shale gas rocks.

The characteristics of a microporosity model comprise of fluids and minerals with different shapes, physical properties and alignments. By mixing different minerals such as quartz, calcite and clays a solid background can be estimated. A dry rock frame is later formed by introducing inclusions into the solid background. The inclusion space may contain inter-particle pores or within pores with clay. The term inclusion here refers to the space occupied by any in-fill material including fluids and solids.

Experiences from various shale gas formations suggest that the modelled results using organic matter treated as inclusion-filling materials generally match well with measured velocity and resistivity log data. However, if we consider the organic matter as the inclusion space, the void spaces are partitioned into solid and fluid volumes (Sone & Zoback, 2011). To attain and determine effective elastic properties of the rock both the substitution, namely fluid and solid substitution must be used.

For the first technique which is the MICP, mercury as a fluid is pumped into the rock to occupy the pore spaces in the rock. The different pressures are then measured with the varying injected volume (Q. Wang et al., 2018). This MICP technique which is used to measure the PSD in shales is said to be inaccurate. This is because there is a limited pore accessibility in shales due to their small pores and thus the mercury injection cannot access these microscopic places. These small and tiny pores also have high capillary pressure and if a

higher pressure of mercury is injected, the shale rock will be compressed and thus the final results will not be accurate.

The second method which is the BET analysis using N<sub>2</sub> and CO<sub>2</sub> gasses will generate a more reliable result. Researchers which have been using the BET analysis found out there are micro-, meso-, and macropores. Using the BET analysis, there is two main process which is the adsorption and desorption of gasses. These two main processes can be used to specify the specified surface area (SSA) and pore volume (Zolfaghari, Dehghanpour, & Xu, 2017).

For , low adsorption is this gas can be useful for characterizing microporosity. Whereas, for adsorption can be used to estimate the meso and macroporosity. Therefore, by combining both the MICP and BET adsorption data, the full spectrum of gas shales can be determined. **Figure 5** shows the PSD using MICP and BET adsorption analysis. By comparing the PSD in the overlapped area (mesopore) it can be seen that the peaks do not match exactly (Wang & Reed, 2009).

The reservoir quality, pore networks and mechanism of fluid flow is very different of shale gas core samples is very different from conventional reservoirs. This is because it has very fine grained particles thus to estimate the complexity is challenging and not very well understood according to some researches (Song et al., 2018). Gas permeability in organic matter which is considerably higher than the permeability of non-organic matrix tends to increase the gas permeability in gas shale because of high porosity, single phase flow and gas slippage effect. For a better and good pathway of high gas production in shales, the natural and hydraulic fractures should be connected. Four different type of porous media are actually present in shale gas. Among them are non-organic matrix, organic matter, hydraulic fracture and natural fractures. Free gas are basically adsorbed and stored in organic matters. In fact a huge amount of free gas is stored in organic matters. **Figure 6** shows the pore spaces which differs from conventional sandstone to gas shale.

#### IV. MINERALOGICAL DESCRIPTION OF SHALE GAS CORE SAMPLES

The following results were obtained by using X-Ray Diffraction. XRD was mainly used to identify the clay minerals and other minerals present in the rock. Figure 1.6 and figure 1.8 illustrates the results for both Kedah and Batu Gajah samples respectively. For the mineralogical content of the Kedah samples, it was seen that it has a very high percentage of quartz (63%), followed by zeolite (33%) and lastly magnesium iron oxide (5%) as shown in **Figures 7, 8**. Figures show that if this sample is to be hydraulically fractured, the performance of the operation would be successful. Zeolite which is a type of clay mineral is also present in the formation. This clay mineral would act as a site for gas adsorption. Magnesium iron oxide is also present in the samples. This would be due to some depositional setting that would be different for every shale rocks. Moving on, for the mineralogical content of the Batu Gajah samples it was seen that it has a very high percentage of muscovite (66%), quartz (29%) and lastly magnetite (5%) as shown in **Figures 7, 9**. This illustrates that these samples have high adsorption sites of gas. Can be easily trapped in clay minerals such as muscovite. On the other hand, a low percentage of quartz are present in this formation. This indicates that if a fracture job would be performed in this region the rocks would perform as well as the Kedah samples. Based on both mineralogical content both samples can be concluded as producing shale samples. No swelling minerals, smectite and bentonite were present in these samples.

Clay minerals which typically consists of kaolinite, muscovite and non-clay minerals such as quartz were seen in the results. Although shale has many other minerals present in it, by using the XRD software it was manageable to determine the minerals. Illinites would normally be found in much older rocks which would be formed after muscovite. Qualitative analysis of XRD was used by employing search and match programmes. This was also increasing challenging due to the material is a mixture of different phases.

#### V. PORE SIZE DISTRIBUTION OF SHALE GAS CORE SAMPLES

Brunauer-Emmett-Teller analysis (BET analysis) was used to obtain the pore size distribution of the shale samples from Kedah and Batu Gajah. Mainly sorption theorems were used to evaluate the model results. The gas sorption thermms are obtained at 77K for the . The BET isotherm results from both shale samples are in **Figures 11, 12**. Which show an increasing the relative pressure the amount of adsorbed gas also increases. Relative pressure consists of two main pressure factors which are the equilibrium pressure and its saturation pressure at the experiment temperature. Desorption themes also exists here as the amount of gas adsorbed decreases by decreasing the relative pressure. This adsorption and desorption term mainly indicates that not all gas which has been adsorbed would be desorbed during the desorption process. This is due to some gas been has been adsorbed onto the organic matter. We can also observe a sharp increase at high relative pressures for both samples. This corresponds to corresponds to capillary condensation at larger pores.

Furthermore, can be classified as type 2 adsorption therm because it exhibits multilayer adsorption. Compared to adsorption which exhibits type 1 isotherm whereby it is known as monolayer adsorption. Therefore adsorption therm would be more accurate in determining the average pore area and volume.

According to Figure 12, the Batu Gajah samples have the highest amount of adsorbed among both the shale samples. The specific surface area (SSA) and pore volume (PV) of the shale samples would eventually impact the amount of gas adsorbed. **Figures 13,14,15** illustrate the BET results for SSA and PV. Batu Gajah samples have higher SSA and PV compared to the Kedah samples. Increasing SSA and PV enhance the capillary condensation during the adsorption process. A higher amount of gas would be adsorbed with the increase of the capillary condensation.

Pore size distribution results are shown in **Figures 15, 16** for Kedah samples and Batu Gajah samples, both show that they have more pores located below 10nm in scale. This proves that shales gas has a permeability of nano-darcies. There were very few and little pores for above 10nm where most of the sample above this scale are all organic matters.

## VI. CONCLUSION

In conclusion for the mineralogical section, both the Kedah and Batu Gajah samples show great representation of shale gas plays. The presence of silicon oxides, quartz and muscovite would be a strong justification for presence of shale formations. Fracturing procedures also could be carried out in these areas because of the available quartz content. The rocks would be able to fracture more easily because of a high brittleness value. Also, noticeable that each mineral has its own fingerprint and no swelling minerals such as smectite or bentonite were found in the minerals.

Moving on for the pore size distribution the BET results shows that both the Kedah and Batu Gajah sample has a majority of pre size distribution of less than 10nm. Batu Gajah also shows a better adsorption of gas molecules. This can be explained by the large pore volume and pore area compared to the Kedah samples. The BET results also shows that adsorption isotherms follows they type 2 isotherms, because it creates an multilayer adsorption.

Finally for total organic content, which shows the total organic carbon content which is useful for hydrocarbon generation. From the results obtained the TOC of Kedah samples shows a higher reading compared to the Batu Gajah samples. This shows hydrocarbon generation would be more feasible from the Kedah samples.

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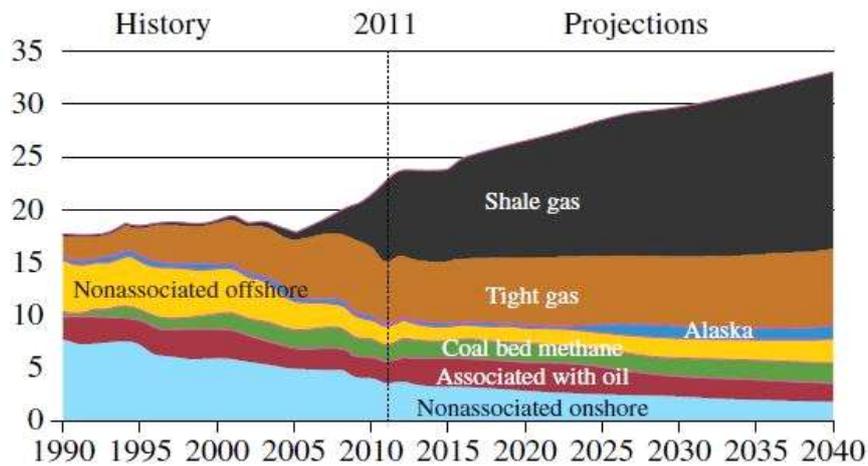


Figure 1: Natural Gas Projections based on Energy Information Administration (Beckwith, 2011)

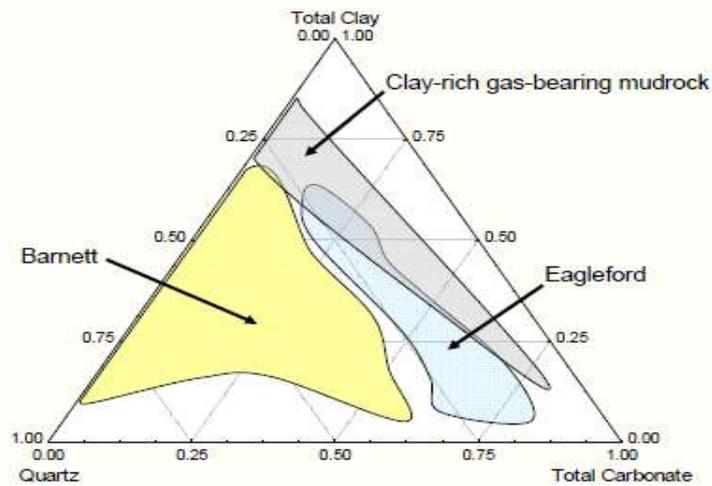


Figure 2: Mineral compositional of shale gas reservoirs (Alyousuf et al., 2011)

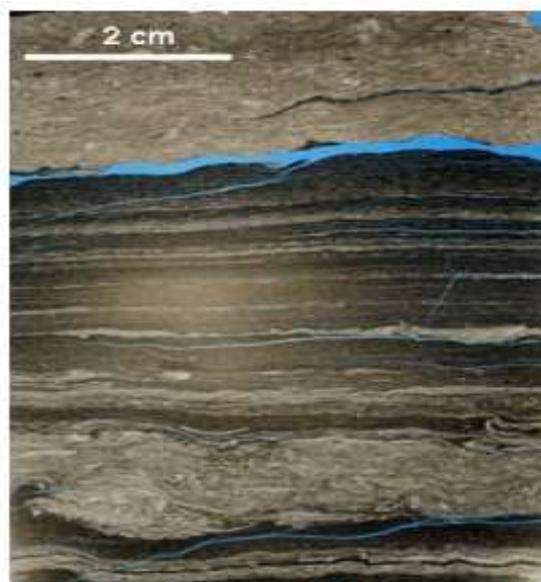


Figure 3 : Shows detailed bedding and composition variations (Passey, Bohacs, Esch, Klimentidis, & Sinha, 2010)

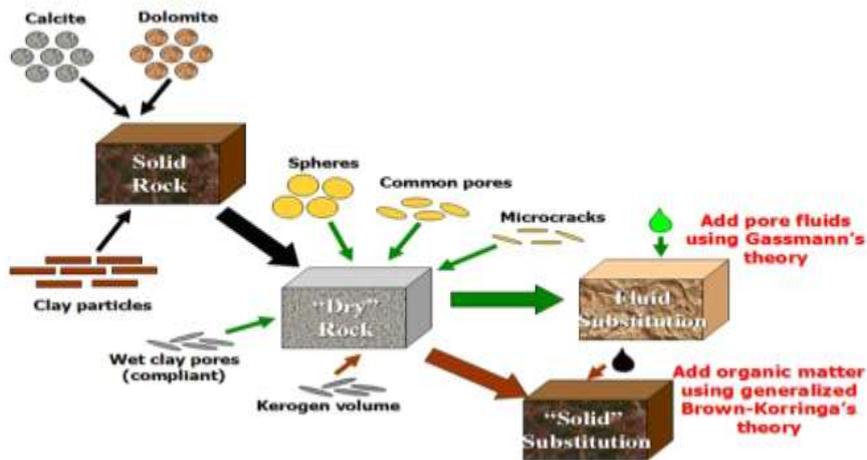


Figure 4: Implementation of rock physics model (Sone & Zoback, 2011)

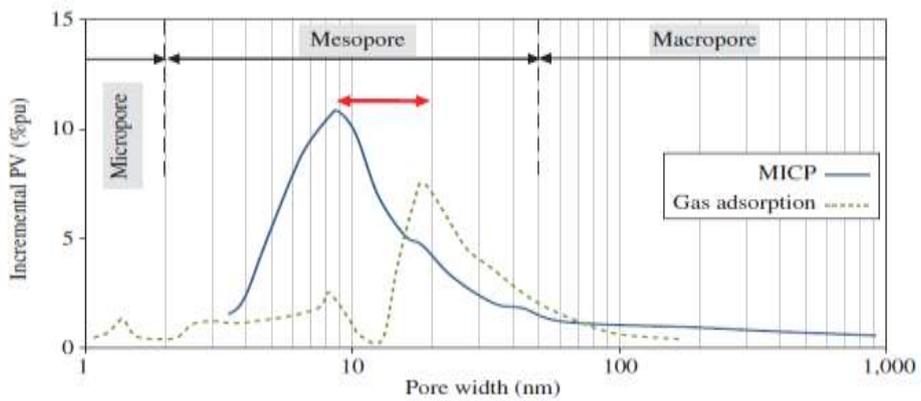


Figure 5: A comparison between MICP and gas adsorption data for shale gas sample (Q. Wang et al., 2018)

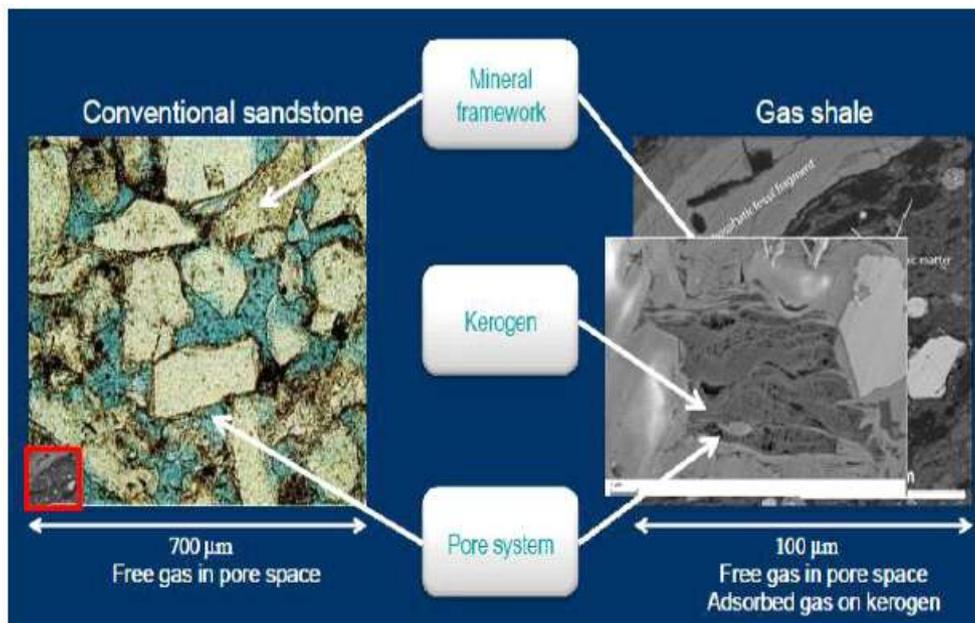


Figure 6: Pore space in sandstone and shale (P. Wang et al., 2018)

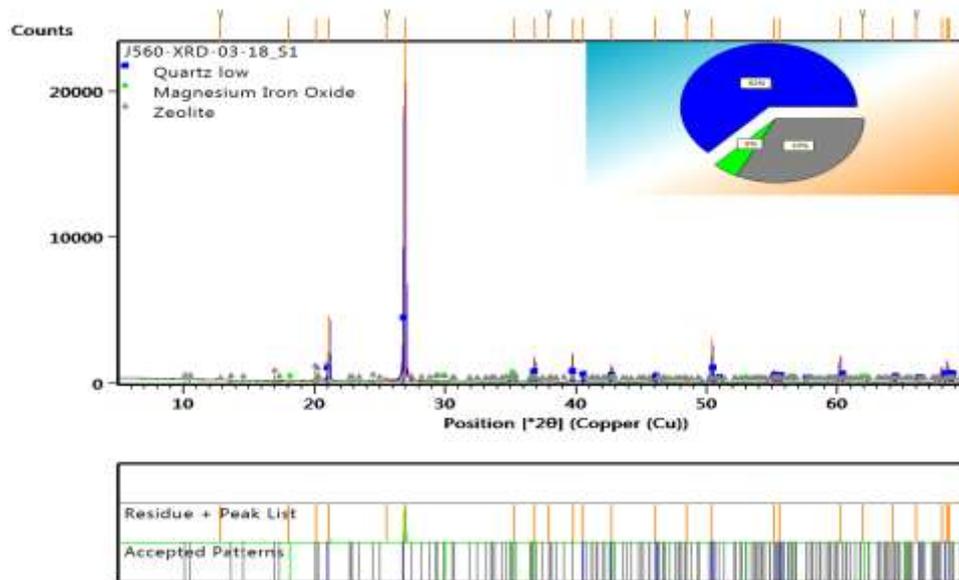


Figure 7: Mineralogical component of Kedah samples

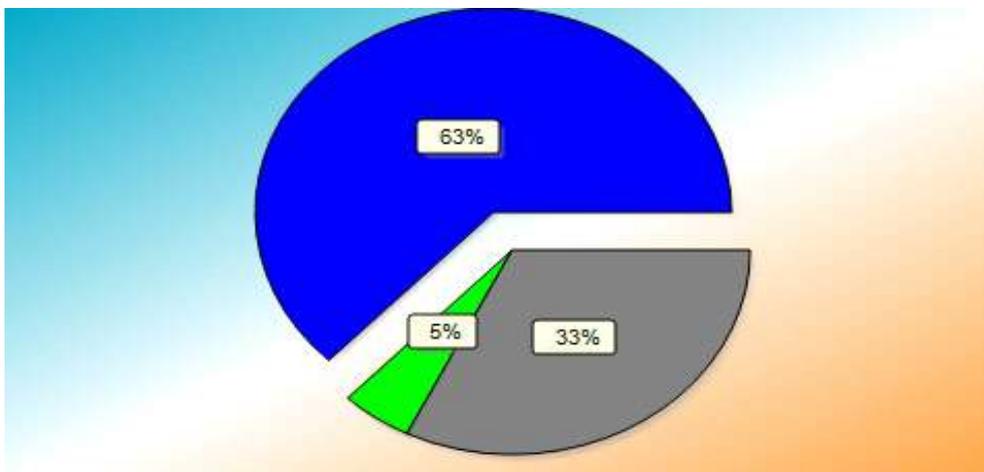


Figure 8: Overall Mineralogical content of Kedah samples

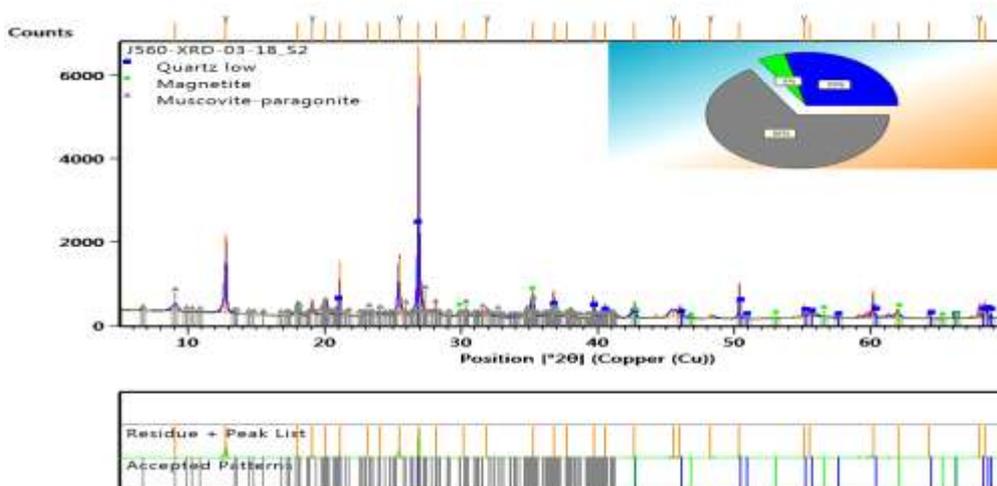


Figure 9: Mineralogical component of Batu Gajah samples

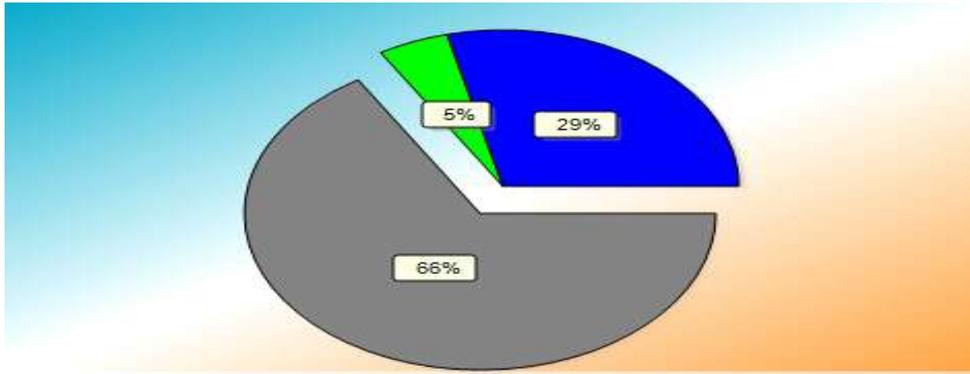


Figure 10: Overall Mineralogical content of Kedah samples

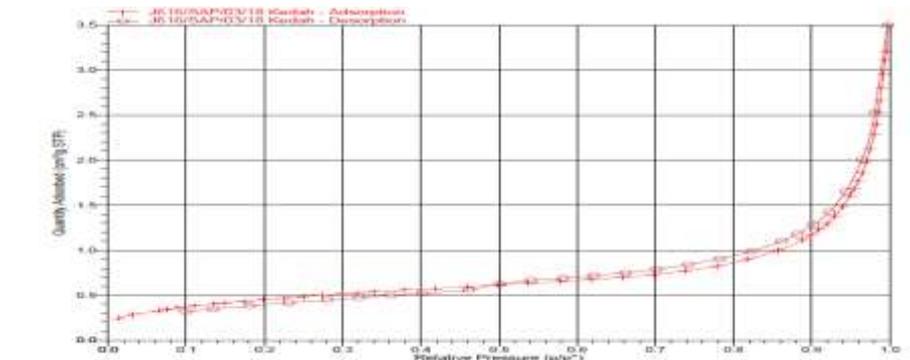


Figure 11: Isotherm results for Kedah sorption threom

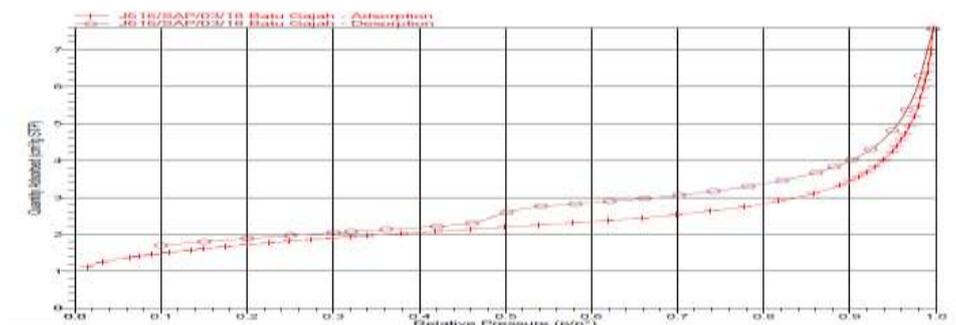


Figure 12 : Isotherm results for Batu Gajah sorption threom

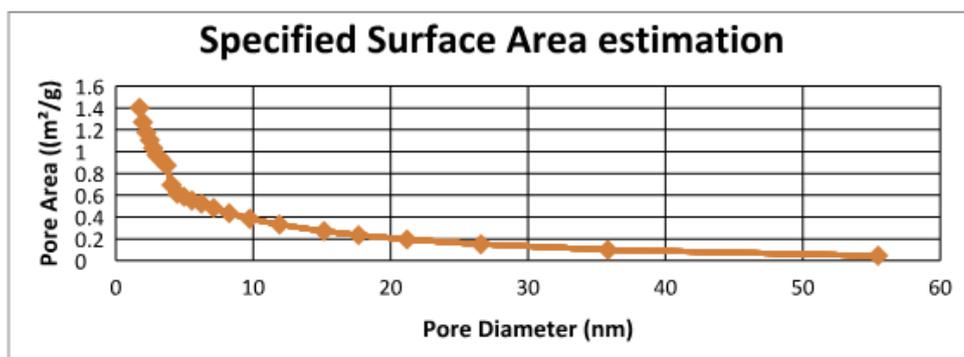


Figure 13: Surface Area Estimation of Kedah Samples

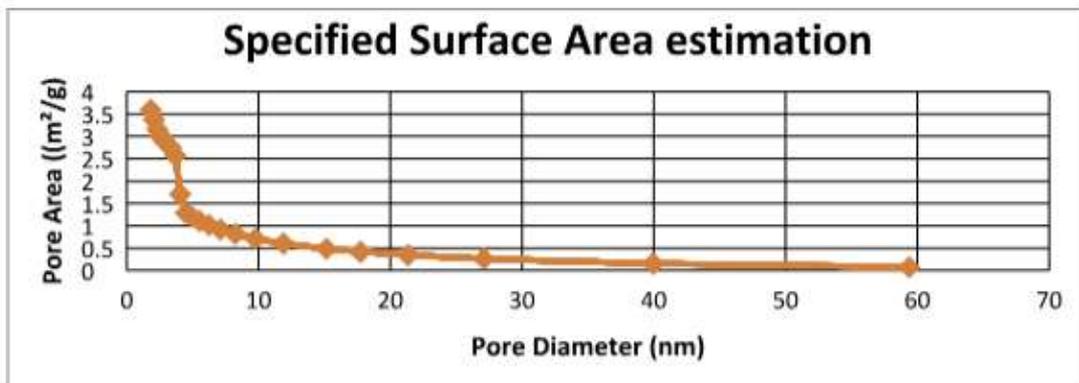


Figure 14: Surface Area Estimation of Batu Gajah Samples

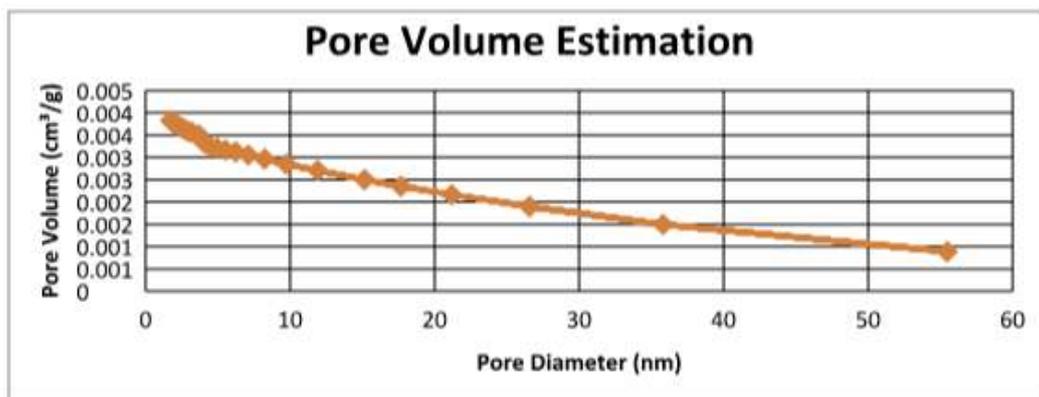


Figure 15: Pore Volume Estimation of Kedah Samples

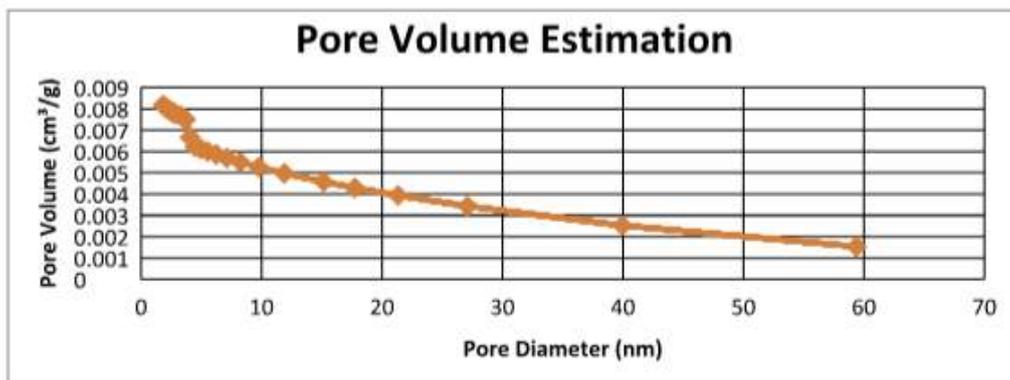


Figure 16: Pore Volume Estimation of Batu Gajah Samples

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