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Palynofacies Analysis and Submicron Pore Modeling of Shale-Gas Plays

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Abstract

The present study combines palynological applications with advanced microscopic techniques to characterize the Utica, Haynesville and Fayetteville shale-gas source rocks. This unprecedented approach could offer an alternative way to measure the total organic carbon (TOC) content using the 2D subsurface Scanning Electron Microscope (SEM) images. This approach is considered to be a faster and inexpensive method compared to conventional geochemical analyses. Palynofacies analysis provided valuable information about kerogen type and its degree of thermal maturation, which are key parameters in shale-gas exploration. Moreover, it qualitatively allowed the estimation of important organic geochemical parameters such as vitrinite reflectance (R_o %) and numerical thermal alteration index (TAI). New high resolution microscopic solutions have successfully been exploited for source rock characterization at both micro- and nano-meter scales. In-situ Focused Ion Beam (FIB) and Scanning Electron Microscope (SEM) technologies provided new insights into rock fabrics such as porosity, permeability, tortuosity, anisotropy and kerogen content. Serial sectioning and sequential imaging using dual beam SEM/FIB instrument were implemented successfully to characterize the 2D kerogen content and 3D submicron-pore structures. Moreover, pores were found in organic matters with the size of nano level and occupy 40−50% of the kerogen body. A successful example of reconstructed 3D pore model from Fayetteville Shale is presented.

Introduction

Qualitative and quantitative characteristics of shale-gas source rock are used by petrophysicists, geochemists and reservoir engineers. These include submicron pore structure and organic matter analyses, which are important in the derivation of rock capillarity, wettability and storativity. Shale-gas source rocks hold large quantities of hydrocarbon reserves that have made significant impact on North American oil and gas market since early 2000s. The ambiguity behind gas storativity and deliverability can be demystified through the understanding of the relationship between organic matter content and porosity.

Conventional standalone analyses are inadequate and not suited for unconventional gas rock characterization. The proposed combined approach provides important information for evaluating and appraising shale-gas plays. Palynofacies analysis identifies intervals of exploratory interest in terms of hydrocarbon content. It can also be used as a proxy to estimate organic geochemical values that are very expensive to obtain and thus, significantly reduces exploration and production costs. Pore imaging and modeling allows the evaluation of gas storage quantity and deliverability in shale-gas plays, which enables the development of optimized processes for hydrocarbon recovery.

Palynofacies analysis as defined by Tyson (1995) is: “the palynological study of depositional environments and hydrocarbon source rock potential based upon the total assemblage of particulate organic matter.” In the present study, palynofacies analysis was carried out on five samples recovered from the Haynesville, Utica and Fayetteville shale-gas source rocks in order to evaluate their kerogen type and degree of thermal maturation. Three of the five studied samples are from the Utica Shale (two samples from Dolgeville member and one sample from the Indian Castle Member), while the fourth and fifth samples are from Haynesville and Fayetteville shales. The data obtained were used to qualitatively estimate some key organic geochemical parameters such as vitrinite reflectance (R_o %) and numerical thermal alteration index (TAI). Samples were also analyzed for TOC content in order to fully understand their source potential.

A key to successfully characterize fluid flow behavior in shale-gas plays is to understand the petrophysics of shale rocks and their submicron-pore structures and kerogen content. A few studies have attempted to construct submicron-pore structures. Liviu *et al.* (2007) conducted an initial intensive study of submicron pore imaging using FIB for chalk rocks. Sondergeld *et al.* (2010) used this method to characterize the submicron structures of a few shale-gas plays. Elgmati *et al.* (2011) have attempted to systematically characterize the petrophysical properties of shale-gas plays. An in-situ dual beam system (SEM/FIB) was exploited for imaging and milling process sequentially. Both 2D organic matter model and 3D structural submicron pore model were reconstructed. Energy-Dispersive Spectroscopy (EDS) was also used to confirm the existence of organic matter and to define rock elements.

Palynofacies Methodology

Conventional palynological processing technique was used to extract the organic matter (kerogen) from the samples. This included crushing about 10−15 grams of the sample in a mortar to the powder size. Samples were then treated with concentrated hydrochloric acid (HCl) for about 24 hours in order to remove their carbonate content. After neutralization, the silicate fraction in the samples was then dissolved by concentrated hydrofluoric acid (HF) treatment for about 72 hours. Thereafter, samples were washed and sieved to remove clay particles and concentrate organic matter. Only kerogen particles that range in size between 10−106 μm were retained to make the final microscopic slides. Slides were then microscopically examined in transmitted light using variable magnification powers for analysis and photomicrography. A total of 500 kerogen particles were counted from each slide. Particles were classified into four main categories namely, structured phytoclasts, degraded phytoclasts, opaques, and palynomorphs. All slides and residues are housed in the Palynology Laboratory at Missouri University of Science and Technology.

Kerogen Type

Kerogen is the dispersed sedimentary organic matter that is resistant to the mineral acids hydrochloric acid (HCl) and hydrofluoric acid (HF) (Tyson, 1993). The classification of kerogen type by Tyson (1993) for routine source rock evaluation was used in the present study, in which kerogen type IV (inert material) was identified from all the studied samples, although they differ in the percentages of individual kerogen components. Kerogen type IV was described by (Peters and Cassa, 1994) as dead carbon, which has little or no hydrocarbon generating capability. It is important to mention, however, that the examined samples (except sample #3 from Utica Shale) likely initially contained kerogen type III (gas prone material) that converted to type IV during the process of thermal over maturation.

The sample from the Haynesville Shale contained abundant very dark brown structured phytoclasts and black opaques in association with frequent very dark brown degraded phytoclasts as shown in **Fig. 1**. Palynomorph-like particles were observed, but could not be confirmed due to their high degree of degradation and very dark color; hence they were counted as phytoclasts. Equant phytoclasts and opaque particles were much more common than lath shaped ones.

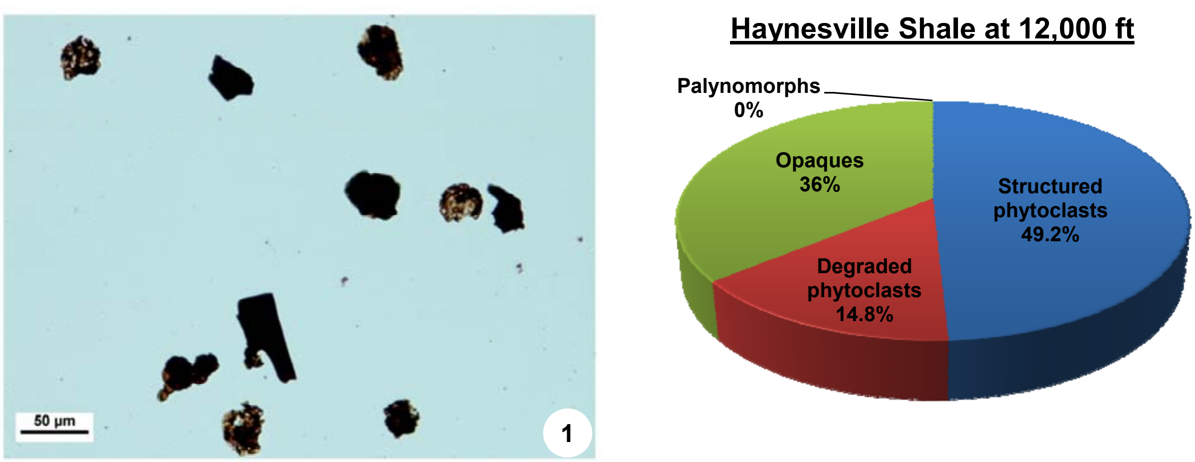


Fig. 1-Percent distribution and photomicrograph of the kerogen components identified from Haynesville Shale which are dominantly phytoclasts and opaques.

Fig. 2 represents the kerogen components that were observed in the shale sample obtained from the Indian Castle Member of the Utica Shale. It shows high abundance of dark to very dark brown structured phytoclasts in association with common black opaques and frequent very dark brown degraded phytoclasts. Palynomorphs (essentially chitinozoans) were very rare and very dark brown to black in color. Many of them were broken down.

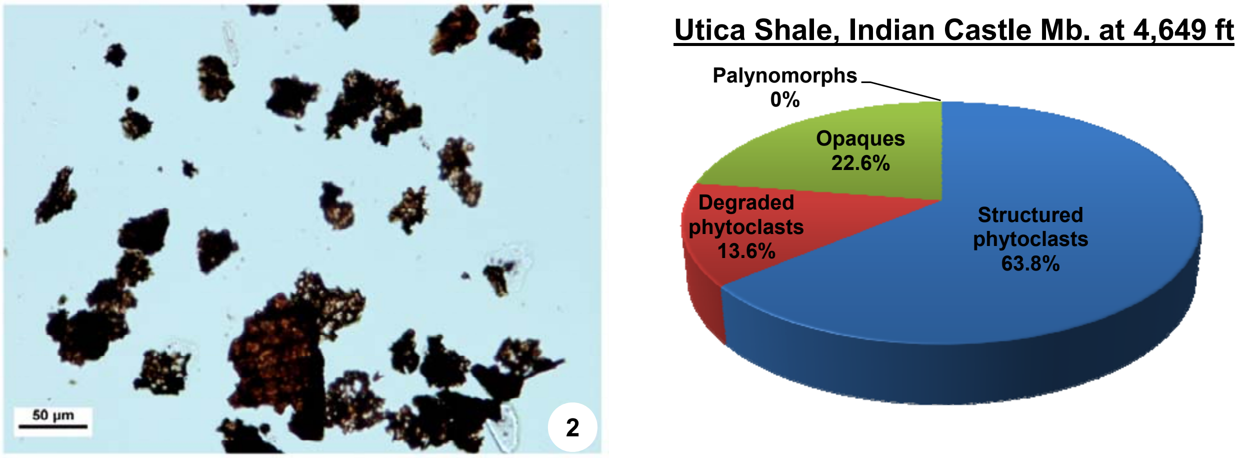


Fig. 2-Percent distribution and photomicrograph of the kerogen components identified from Utica Shale, Indian Castle Member which are dominantly structured and degraded phytoclasts.

Samples from the Dolgeville member of the Utica Shale were not similar in their kerogen composition. The first sample at the depth of 4,878 ft, revealed an overwhelming abundance of black opaques with rare dark brown structured phytoclasts as shown in **Fig. 3**. The majority of opaque particles were equant in shape and smaller in size than those recovered from other samples. Very rare palynomorphs were spotted (also essentially chitinozoans) and they were very dark brown in color and extremely degraded and/or broken down.

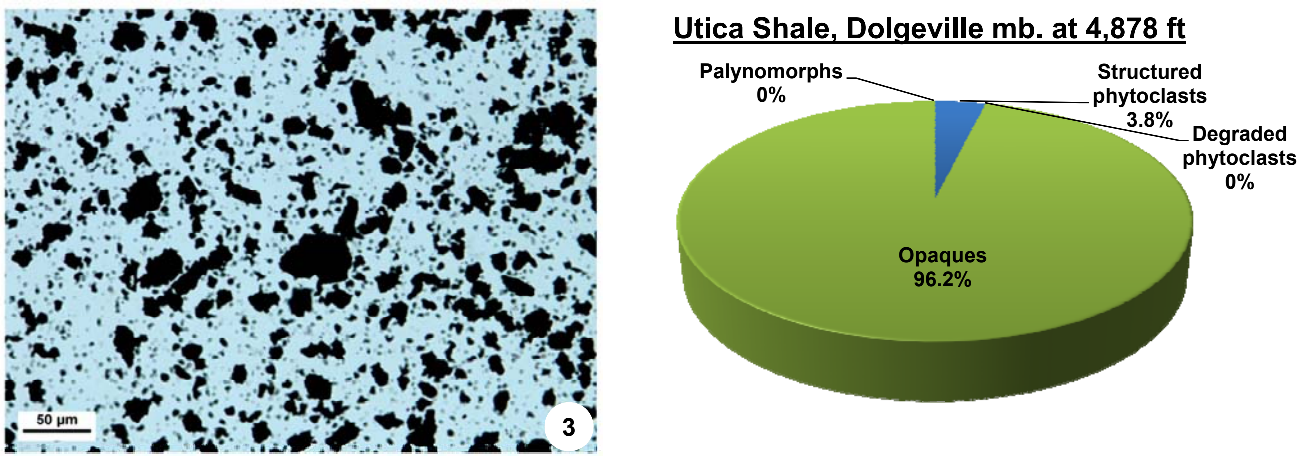


Fig. 3-Percent distribution and photomicrograph of the kerogen components identified from Utica Shale, Dolgeville member at the depth of 4,878 ft which are dominantly opaques.

The second sample of Dolgeville member at the depth of 5,197 ft, on the other hand, contained high abundance of very dark brown degraded phytoclasts along with common black opaques and frequent dark to very dark brown structured phytoclasts as illustrated in **Fig.4**. Palynomorphs were very rare compared to the total kerogen composition. Palynomorphs (essentially chitinozoans) were highly degraded and very dark brown to black in color.

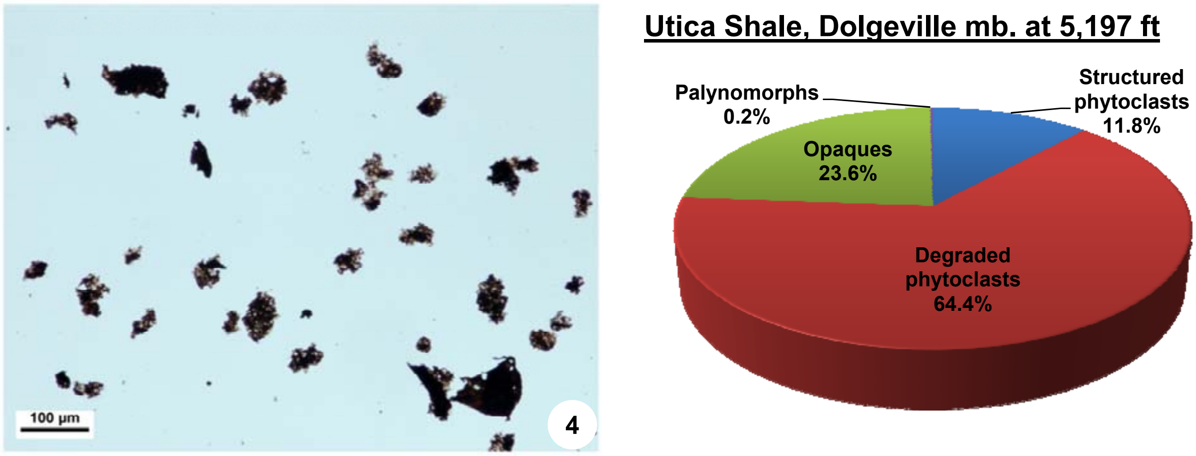


Fig. 4-Percent distribution and photomicrograph of the kerogen components identified from Utica Shale, Dolgeville member at the depth of 5,197 ft which are dominantly degraded phytoclasts.

The fifth shale-gas sample acquired from Fayetteville Shale play had very high abundance of black opaques in association with very little structured and degraded phytoclasts as presented in **Fig. 5**. No palynomorphs were observed during the counting process. Almost all of the kerogen particles in this sample were equant in shape.

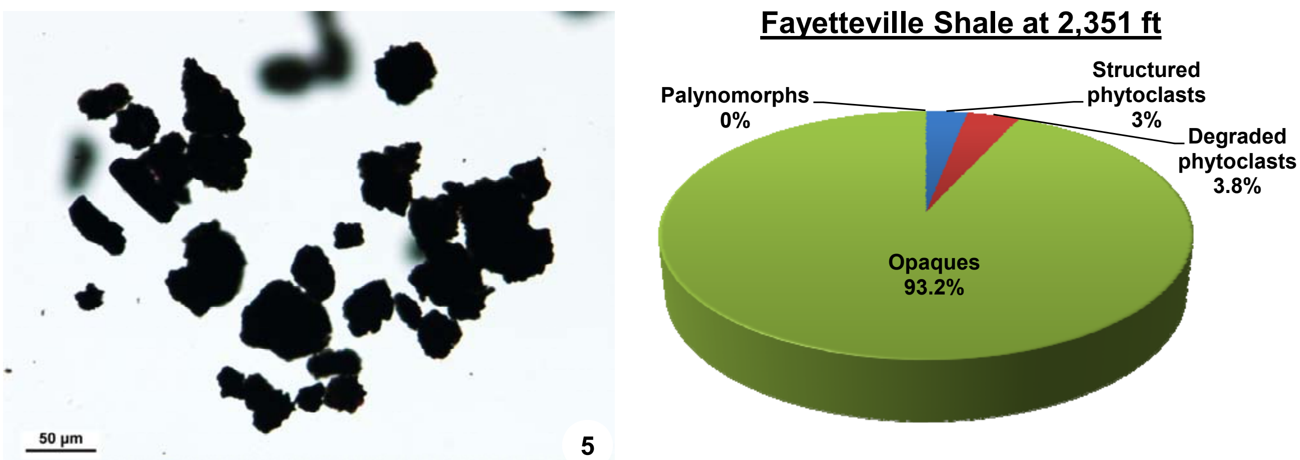


Fig. 5-Percent distribution and photomicrograph of the kerogen components identified from Fayetteville Shale which are dominantly opaques.

Fig. 6 shows images of some chitinozoan specimens which were observed in the examined shale-gas samples. Their wall colors are ranging from dark brown to nearly black indicating post-mature thermal phase.

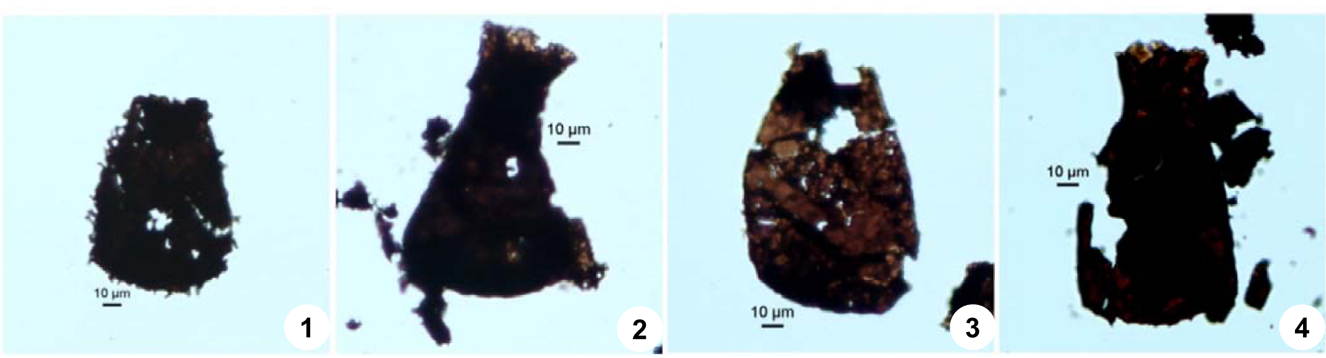


Fig. 6-Photomicrographs of some of the chitinozoan specimens identified from Utica and Haynesville shale-gas source rocks.

Thermal Maturation

Thermal maturation is the chemical change induced by post-depositional heating over time that transforms sedimentary organic matter into hydrocarbon (Peters and Cassa, 1994; Traverse, 2007). Source rocks are thermally classified into immature, mature, and post-mature depending on the temperature level they were subjected to (Peters and Cassa, 1994).

Color changes in the exine of fossil palynomorphs (e.g. spores and pollen) have long been used to interpret the degree of thermal maturation of potential source rocks. In the present study, colors of the pseudochitinous walls of chitinozoans and the exine of palynomorph-like grains (gymnosperm pollen?) recovered from the Utica and Haynesville shale-gas samples, respectively were examined in order to visually estimate their thermal maturation. The sample from Fayetteville Shale was excluded, since no palynomorphs were observed. Although chitinozoans and spores/pollen are different in their wall composition, their rates of color changes in response to thermal maturation are almost identical (Traverse, 2007). Observed colors were matched to the spore/pollen color chart which can be found in Traverse (2007, p. 584).

All palynomorphs in the examined samples were dark to very dark brown in color, sometimes even black, implying high level of maturation (i.e. post-mature source rocks) as depicted in **Fig. 6**. This denotes that the organic matter have already generated the hydrocarbons respective to their kerogen type. This signifies that the shale-gas sample from Dolgeville member at the depth of 4,878 ft (inert kerogen material) has generated little dry gas, or nothing, while those of other samples (with initial kerogen type III content) have generated wet gas and condensate (cf. Batten, 1980). Since all the samples currently contain thermally post-mature type IV kerogen, their source potential is limited to minor amounts of dry gas, or barren, at the present time.

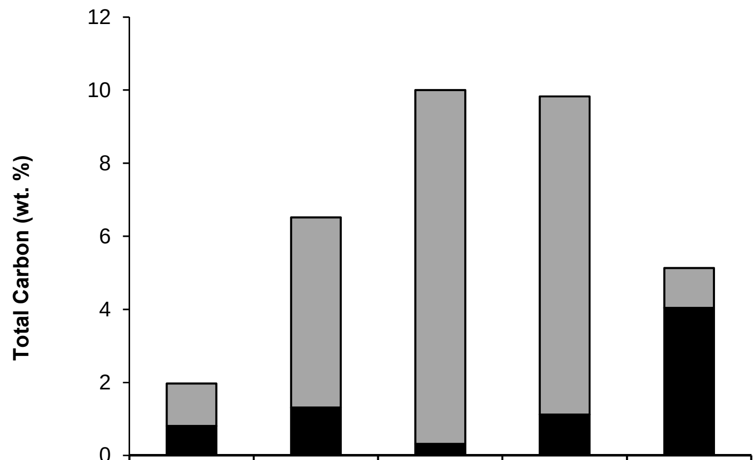
Estimated Key Geochemical Parameters

Several studies have demonstrated the strong correlation between data obtained from palynofacies analysis and those from instrumental geochemical analyses (e.g. Zobaa *et al.*, 2007, 2009; El Beialy *et al.*, 2010). Detailed list of the pros and cons of both methods in organic maturation studies can be found in Brooks (1981). However, the relatively inexpensive nature of palynofacies analysis makes it powerful in preliminary exploratory studies limited by tight budgets. The aforementioned data about kerogen composition and thermal maturation has been employed to qualitatively estimate some key geochemical parameters, such as numerical TAI and vitrinite reflectance (R_o %). The dark to very dark brown colors of palynomorph walls in the studied samples (excluding Fayetteville Shale sample), which are typical post-mature source rocks, correspond to 4- to 4 TAI and ~1.5–2.5% vitrinite reflectance (Traverse, 2007). This further suggests that these source rocks are mainly in the metagenesis thermal alteration stage (dry gas zone) indicative of about 150–200° C temperature range (Peters and Cassa, 1994).

Total Organic Carbon (TOC)

The studied samples were quantitatively investigated in the laboratory for TOC analysis. Samples were grounded and weighed into tin capsules and combusted in a Carlo Erba EA at 1000° C to measure the total carbon content. Samples were then weighed out again into exetainer tubes and acidified with an AutoMate Prep Device. Evolved CO_2 was measured using a UIC 5011 coulometer in order to calculate the total inorganic carbon. TOC is the weight percentage of the difference between total carbon and inorganic carbon.

When interpreting TOC data, circumspection should be considered because it is not a direct measure of hydrocarbon source potential. Instead, a combination of several proxies in addition to TOC must be considered (e.g. lithologic composition, sedimentation rate, kerogen type, thermal maturation, basin redox conditions, etc.). The TOC for a given source rock is composed of three fractions, namely organic carbon in the extractable hydrocarbons, organic carbon that can be transformed into hydrocarbons, and dead carbon that cannot yield hydrocarbons (Jarvie *et al.*, 2007). This clearly implies that TOC content will diminish in a source rock with hydrocarbon expulsion. The analyzed samples have TOC contents of 0.31–4.04 wt. % as shown in **Fig. 7**. It is strongly possible that these rocks have once held more TOC content that has decreased over time through the process of thermal maturation and hydrocarbon generation and expulsion. Therefore, it is likely that most of the TOC, at present, is dead carbon. Accordingly, the studied rocks are believed to have lost their hydrocarbon producing capabilities, even though their TOC is higher than the accepted minimum (0.4 wt. %) for source rocks under appropriate conditions. High inorganic carbon content was also observed in Utica Shale samples, which is likely resulted from high concentrations of calcite ($CaCO_3$) in this shale-gas play as indicated by Elgmati *et al.* (2011).



	#1	#2	#3	#4	#5
Inorganic C	1.16	5.21	9.69	8.71	1.09
Organic C	0.81	1.31	0.31	1.12	4.04

Sample #1 : Haynesville Shale at 12,000 ft

Sample #2 : Utica Shale, Indian Castle Mb. at 4,649 ft

Sample #3 : Utica Shale, Dolgeville mb. at 4,878 ft

Sample #4 : Utica Shale, Dolgeville mb. at 5,197 ft

Sample #5 : Fayetteville Shale at 2,351 ft

Fig. 7-Measured total carbon (inorganic and organic) from the studied shale-gas source rock samples.

3D Submicron Pore and 2D Organic Matter Modeling

Submicron pore imaging and modeling provide insights into the petrophysical properties of shale-gas source rocks such as pore size histogram, porosity, tortuosity, anisotropy, and TOC. A dual beam system (SEM/FIB) was utilized to reconstruct the 2D kerogen model and the 3D structural pore model of shale-gas plays. The detailed procedure of serial sectioning and sequential imaging using dual beam SEM/FIB instrument proposed by Elgmati *et al.* (2011) was implemented successfully to characterize submicron pore structure. In which, two-hundred 2D SEM images were used to reconstruct the original 3D submicron-pore structure along with the organic matter (Kerogen) and the inorganic material (e.g. clay platelets). It is worth mentioning that this distinct approach may offer an alternative way to measure the TOC content from 2D SEM images. It is a faster and inexpensive method compared to conventional geochemical analysis. A successful example of reconstructed submicron pore model from Fayetteville shale-gas sample is presented. Furthermore, Energy-Dispersive Spectroscopy (EDS) was used to affirm the existence of organic matter and to identify the elemental composition of the examined samples.

After performing serial sectioning and sequential imaging of 200 images from the Fayetteville Shale sample, all the 2D SEM images were stacked into 3D pore structural model. The process was performed through two main steps. The first involved creating a 2D SEM model of the kerogen nano pores as shown in **Fig. 8**. This was then translated into a 3D model. Commercial imaging softwares were used for reconstructing the 3D model and obtaining and visualizing advanced qualitative and quantitative information from the shale-gas SEM images. The original image (A) was converted into binary image (B) with pixel values of 0 and 1.

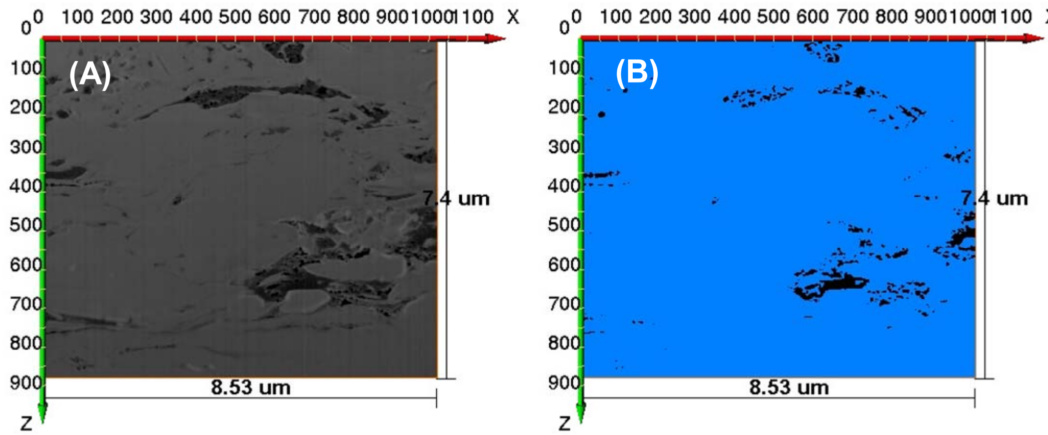


Fig. 8-2D kerogen pore model of Fayetteville Shale at depth of 2,351 ft; (A) SEM images showing the organic matter, and (B) Converted 2D binary image of 0 and 1 pixel values.

Element anaylsis was performed at three positions as shown in **Fig. 9**. According to spectrum counts, the dark porous spots represent kerogen materials which contain high organic carbon contents. Meanwhile, the solid part is believed to represent aluminum silicate class mineral (possibly illite). The extracted TOC value from the previous 2D SEM image shown in **Fig. 8** is 3.91%. It highly matches the obtained TOC from conventional geochemical analysis which was 4.04 wt. %. This method may be used for future studies as an alternative way for measuring TOC content in shale-gas rocks. It is fast and relativly inexpensive.

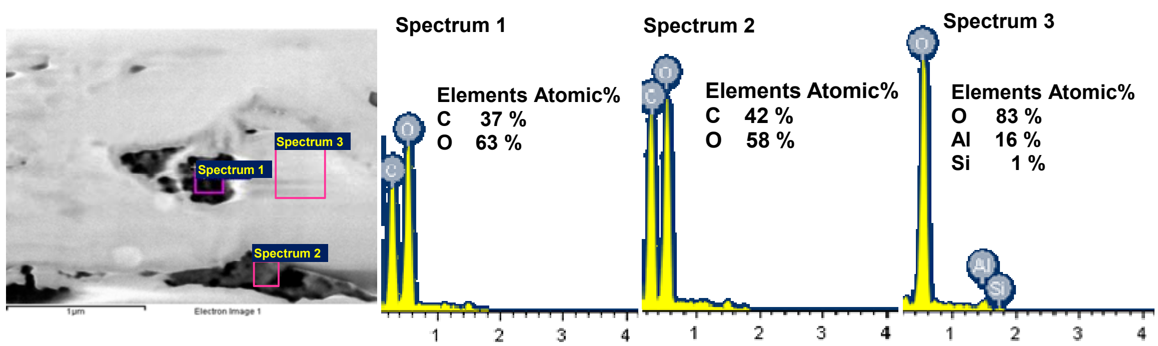


Fig. 9-EDS spectrum analysis of Fayetteville Shale at depth of 2,351 ft; spectrum 1 and 2 confirm kerogen presence, and spectrum 3 represents aluminum silicate (clay).

A scale transformation from pixels into micrometers is done according to image size and magnification scale. A 2D quantification module was utilized to handle the quantitative analysis of kerogen pore size calculation and TOC content. However, this algorithm also works for micron-sized pores and fractures within the shale body. The resulted histogram of 2D SEM model in **Fig. 10** presents micron-sized pores as the major pores. The extracted rock porosity is 3.34% and kerogen porosity occupies about 40−50 % of organic matter. Nano pores in kerogen are between 10−50 nm. The derived kerogen permeability is 4.76×10^{-4} md using average pore throat diameter of 30 nm. Rock permeability (k) is determined by using the adjusted Kozeny equation which takes pores tortuosity into account as shown in the following equation:

$$k = \frac{\phi\, d_p^2}{32\,\tau} \qquad (1)$$

Where ϕ is the rock porosity, d_p is the average (mean volume) diameter of the pores and τ is the tortuosity coefficient. It is assumed that tortuosity is equal to 1 in the 2D kerogen model.

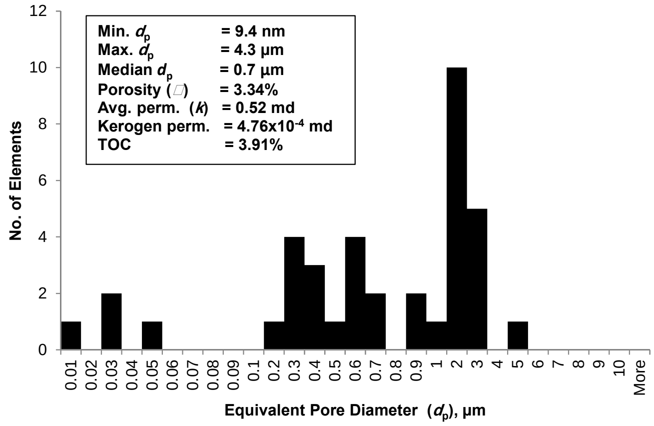


Fig. 10-Fayetteville Shale kerogen pore size histogram resulted from 2D pore model.

The second step, before stacking the images, involved the alignment process that was done at a marked feature on the image in order to eliminate the mechanical and beam drifts. Then, 200 slices were loaded into a 3D bounding box. A representative elemental volume of 8.85×8.65×9.62 µm was extracted from the bounding box. It is believed that the representative elemental volume is able to provide key insights into the petrophysical properties of the shale gas sample. Optional smoothing and filtration algorithms may be applied if required. Image voxels were converted into a meaningful scale (µm) by taking into account of the original image's magnification scale. Binary image conversion and separation were also required within the chosen voxel thresholds by using some quantification modules which are associated with the stacking software. It is used to determine grain element boundaries for defining the pore structure of shale-gas. **Fig. 11** summarizes all these steps.

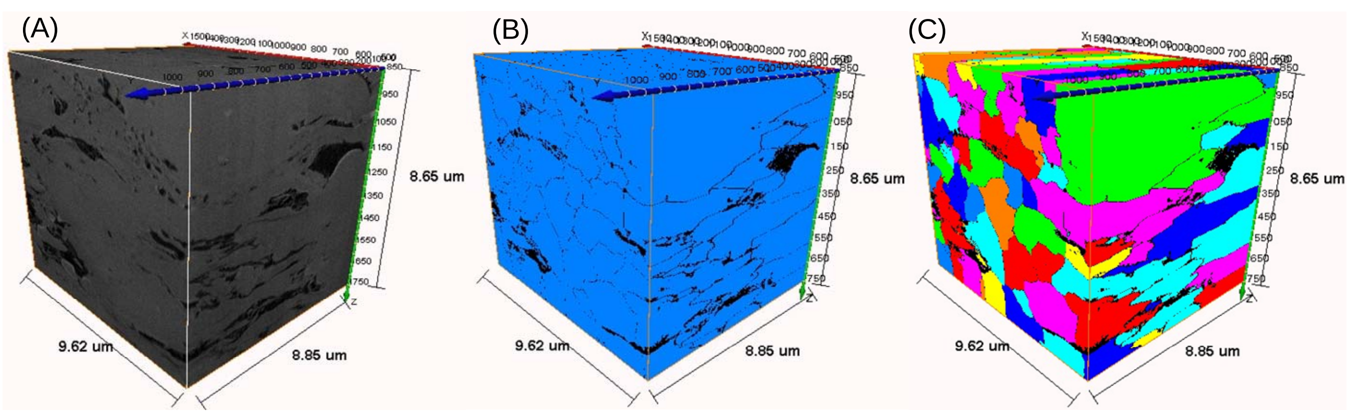


Fig. 11-3D pore model of Fayetteville Shale (A) 200 slices of 2D-SEM images are aligned and stacked, (B) Converted 3D stack to binary image of 0 and 1 voxel values, and (C) Element boundaries are determined and labeled to perform the porosimetry analysis.

Once all the previous steps are carried out, an analysis module is used to perform statistical analysis and to calculate rock pore size distribution according to the chosen separation algorithm. Moreover, block porosity, tortuosity and anisotropy were produced. As seen from **Fig. 12**, the major pore size, which corresponds to the highest frequency, is 30 nm. This validates the analysis of 2D kerogen model. Only a small number of micron-sized pores exist in this 3D model.

Tortuosity coefficient (τ) is basically defined by the ratio of actual flow path (L_a) to the total sample length (L). It is always greater than 1 for heterogeneous rocks. From most rock laboratory standpoint, tortuosity can also be defined by the following equation:

$$\tau = \left(\frac{L_a}{L}\right)^2 \qquad (2)$$

Using anisotropy definition of vertical to horizontal permeabilities ratio (i.e. $\beta = k_v/k_h$) and assuming that horizontal permeabilities (k_x and k_y) in x and y directions are equal to k_h, both vertical and horizontal permeabilities were determined.

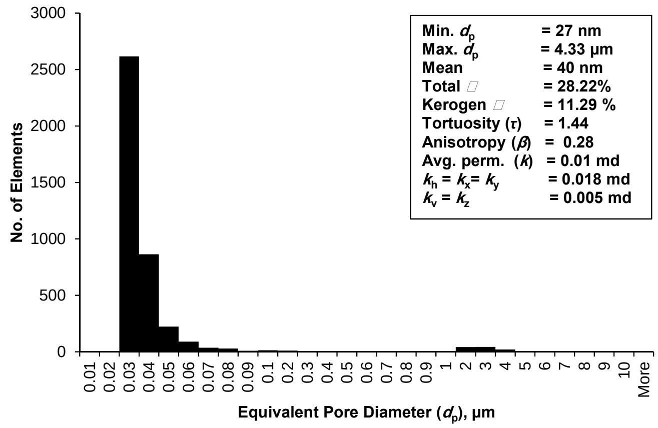


Fig. 12-Fayetteville Shale pore size histogram resulted from 3D pore model.

Submicron pore modeling is believed to be a powerful tool to characterize the petrophysical properties of shale-gas source rocks. It provides a direct measurement of the original pore structure and it defines the bulk pores of the shale sample without any manipulation. It also offers a new quantitative approach to determine the total organic carbon content (TOC). There is about 3% error in the computed TOC from the 2D SEM image. However, the diagnosis scale is in submicron scale compared to the gigantic scale of the other measurements. In order to make results universal, a comprehensive statistical study is required.

Conclusions

The following conclusions are drawn from this study:

1) Kerogen type IV (inert material) was identified from all the studied samples, although they differ in the percentages of individual kerogen components. The examined samples (except sample #3 from Utica Shale) likely initially contained kerogen type III (gas prone material) that transformed into type IV during the process of thermal over maturation.

2) The observed palynomorphs in the palynomorph productive samples implied high level of maturation (i.e. post-mature source rocks), which denotes that the organic matter have already generated the hydrocarbons respective to their kerogen type. This signifies that the shale-gas sample from Dolgeville member at the depth of 4,878 ft (inert kerogen material) has generated little dry gas, or nothing, while those of other samples (with initial kerogen type III content) have generated wet gas and condensate. Since all the samples currently contain thermally post-mature type IV kerogen, their source potential is limited to minor amounts of dry gas, or barren, at the present time.

3) Measured TOC content ranged from 0.31−4.04 wt. % in the studied samples. It is likely that they once contained more TOC content that has decreased over time through the process of thermal maturation and hydrocarbon generation and expulsion.

4) Pores of organic matters were found in nano size and occupied 40−50% of the kerogen body. Our study proved that the petrophysical properties of the original pore structures can be effectively computed from reconstructed three-dimensional models.

5) Good agreement between the computed TOC from the 2D SEM image and the measured TOC in the laboratory with an approximate error of 3%. This encourages exploiting 3D submicron porosimetry and kerogen content for future studies.

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