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RESEARCH ARTICLE

The Role of Fractal Micro-Pore to Absorption of Methane Gas, Case Study: Coal of Tanjung Formation, Arang Alus Area, Banjar District, South Kalimantan, Indonesia

Sugeng Raharjo[1*], Basuki Rahmad[1], Ketut Gunawan[2], Budi Prayitno[3]

[1] *Geological Engineering Universitas Pembangunan Nasional Veteran Yogyakarta, Indonesia*
[2] *Mining Engineering Universitas Pembangunan Nasional Veteran Yogyakarta, Indonesia*
[3] *Department of Geological Engineering, Universitas Islam Riau, Riau, Indonesia*

*Corresponding author : sugengrhj@upnyk.ac.id
Tel.: +62 812-2607-1611
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Abstract

The Tanjung Formation is one of the coal bearing formations in the Barito Basin, South Kalimantan. The coal seams in the Tanjung Formation in the Arang Alus area have 4 (four) seams,there are seam A, B, C, and D. The age of these coal seams are Eocene - Oligocene with a thickness between 0.5 - 2 meters. This study aims to determine the characteristics of micropore fractal and methane gas absorption from coal samples taken by channel sampling on exposed coal in the open pit. The method used is SEM analysis, vitrinite reflectance ($R_{o,max}$), adsorption isotherm, and fractal calculation. The four coal seams based on vitrinite reflectance values (0.52 %- 0.62 belong to the sub-bituminous rank. Based on the methane gas absorption capacity for coal seam C of 450 SCF/ton while coal seams A, B and D of 308 SCF/ton, 336 SCF/ton and 407 SCF/ton, the fractal pore dimension value in seam coal C = 1.963 is higher than seam coal A = 1.933, B = 1.940 , and D = 1.943. The small size of the fractal pore dimension value caused by the degree of regularity of the micropore distribution in each coal seam methane differences.

Keywords: Vitrinite Reflectance, Adsorption, Fractal, Micropore, Methane Gas

1. Introduction

The research area is located in the Arang Alus area, Banjar district, or 60 Km from Banjarmasin, Indonesia, towards the north. This location was revealed by coal seams found in the Tanjung Formation. Tanjung coal has a thickness of 50 cm to 300 cm. Coal in the Tanjung Formation is a coal-bearing formation in South Kalimantan, Barito basin. The coal seams in this formation has 4 (four) main seams; there are seam A, B, C, and D. The age of the Tanjung Formation is Eocene – Oligocene (Heryanto, 2009). The rank of coal in this formation is classified as bituminous. Megascopically, Tanjung Formation coal has shiny black (bright banded), conchoidal, and light fractions.

Coal is a methane gas reservoir rock, storing gas mainly by adsorption on the surface of pores. The structure of coal pores, including the pore shape, pore distribution, pore size, and pores interconnected, determine the porosity and permeability of coal, further affecting the gas absorption capacity and gas volume (Zhang & Li, 1995); (C. J. Liu, Wang, Sang, Gilani, & Rudolph, 2015). The success of methane gas exploitation is highly dependent on the heterogeneity of the coal pore structure. Therefore, understanding the characteristics of coal adsorption and pore structure is very important to predict the size of the gas volume in the exploration of Coal Bed Methane.

Pore structure characteristics are influenced by coal type and coal rank, which are two interdependent factors (Clarkson & Bustin, 1996) Based on pore size in coal divides into: micropore (diameter < 2 nm, mesopore (2 nm < diameter > 1000 nm, and macropore (diameter > 1000 nm). The size of the pores has a different absorption effect on the absorption capacity of methane gas. In general, micropores and mesopores are the main space for the absorption of methane gas.

Fractal geometry, first created by (Mandelbrot, 1982) has proven to be a proper analysis of materials with irregular pores and rough surfaces, including coal (Friesen & Mikula, 1987); (Pyun & Rhee, 2004). Fractal geometry analysis can be used to determine the relationship between pore or surface structure and the absorption capacity of methane gas. The fractal theory is an effective method for characterizing pore structure heterogeneity. However, this research was previously carried out by several experts using various methods, including Scanning Electron Microscope (SEM) (Pyun & Rhee, 2004), Transmission Electron Microscopy (TEM) , Nuclear Magnetic Resonance (Wang, Cheng, Lu, Jin, & Zhao, 2014), and porosimetry of mercury (Cuerda-Correa, et al., 2006). All of the above methods are used to investigate fractal characterization from porous media. However, only the mercury porosimetry method cannot get accurate information about the micropore. The mercury porosimetry is used to characterize mesopores and macropores. Therefore, methane gas adsorption analysis was used to characterize micropores and mesopores. Based on the physical description of fractal pore dimension and pore structures, research by (Cuerda-Correa et al., 2006) and (Yang, Ning, & Liu, 2014) concluded that micropores have a more significant influence on fractal pore dimension than mesopores and macropores. The calculation for gas absorption is using the Langmuir equation. Several experts have applied these calculations before calculating coal methane gas absorption, which is considered a monolayer (Gregg & Sing, 1982). Although coal methane absorption is not a monolayer, Langmuir's model can still be applied. The coal has the type of adsorption isotherm. Therefore, it is necessary to conduct a more detailed study in the exploration of coal methane gas. This study aims to determine the relationship of fractal pore dimension to the behavior of coal methane absorption. This research was conducted on coal, which has almost the same rank. This study and the results can help to understand the pore system and absorption of methane gas in coal exploration in Indonesia. The research location is in the Arang Alus area, Tapin Regency, South Kalimantan Province, Indonesia.

2. Method

Primary data in this study were collected from field data from four sampling locations, and it is represented by coal seam A, B, C, and D. The sampling locations can be seen in the following table (Table1). Coal sample data were taken using the method channel sampling from top to bottom of each coal seam. Each coal seam correlated with outcrop at coordinates 292858,693 meters East and 9638682,718 meters North (Figure.1). Coal samples were dried at 40˚C then crushed, then samples were taken using a reliable 250 grams method, then sieved with 1 mm particle for coal petrographic and 0.012 mm for isotherm analysis.

Table 1. Location sampling coordinate

Seam Coals	m E	m N
A	294610	9638026
B	294612	9638039
C	294613	9638040
D	294620	9638085

The samples resulting from sieving 0.6 - 1.0 mm, were used to make polishing sections with the Meta Serv 250 tool, standard observation procedures. Vitrinite reflectance was measured using the Craic Coal Pro microscope. The procedure to determine vitrinite reflectance is to standardize the sample first with the vitrinite reflectance measurement standard in the microscope: Spinel = 0.427, Sapphire = 0.505, N Last 46 A = 1.37, after standardizing the vitrinite reflectance and then observing the magnitude of the vitrinite reflectance.

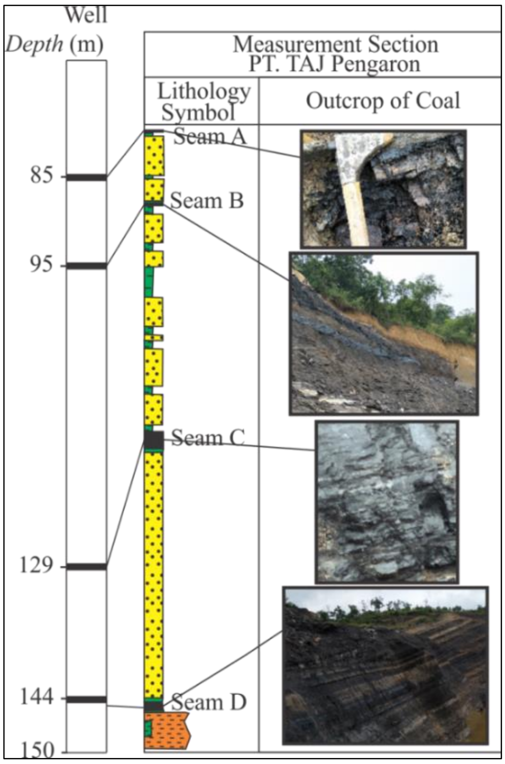

Fig 1. Well log correlation using outcrop and number sampling

The Isotherm Adsorption Test requires a weight of 250 grams of the sample, the sample is crushed with a crusher to form a grain-sized powder that passes through the screen 0.121 mm (80 mesh) opening. The initial isotherm adsorption test process uses ASTM D1412-85. The sample reconditioning must weigh and place the sample in the decicator below given a K2 SO4 solution then in a vacuum condition conditioned at 300 C. The adsorption isotherm test is carried out based on the volumetric method to determine sorption capacity as a function of pressure; the gas used is methane gas (CH_4) purity 99.9%.

The volumetric method refers to Australia's Commonwealth Scientific and Industrial Research Organization (CSIRO). In this method, the volume of gas absorbed by the sample is measured indirectly by injecting methane gas gradually with pressure varying to 16 Mpa (2320 psi) with varying temperatures. This test kit is operated automatically via a computer with the software Adsorption Isotherm System (CSIRO) so that the pressure when injection can control.

The relationship of volume - pressure at a certain temperature (sorption isotherm) can be used to determine the gas storage capacity and estimate the volume of released gas from the sample in line with the decrease in reservoir pressure. In general, the relationship between storage gas capacity and pressure uses the Langmuir equation:

$$Gs = \frac{(VL\ P)}{(PL+P)} \ldots\ldots\ldots\ldots\ldots\ldots\ldots 1$$

Where: Gs = Storage gas capacity, m3 / ton
P = Pressure, KPa
VL = Langmuir Volume Constant, m3 / ton
PL = Langmuir pressure constant, KPa

At the image processing stage, the sample is analyzed with SEM. The sample is scanned with a relatively large current source of 50 µA, the source of the voltage is 60 kV, the lighting time is 8 seconds. The image generated from the scanning process is a digital image and image from the grayscale scale sample. The next process is image processing using Matlab software.

This process distinguishes between solids and pores of coal by changing a gray image into a binary image, and then thresholding is performed. This binary image serves to distinguish between black pores and the edges of white granular solids. Each black pore boundary area values 0 pixels (black), and a stable pore border value is 254 pixels (white). The fractal pore dimension calculation uses the box-counting method (Mandelbrot, 1982). The usual dimensions are denoted by D, which states each object's topological dimensions of a fractal. The resulting number of sub-segments from the iteration of a fractal object is denoted by N, while the length of the subsidy denoted is by r. So the relationship between D, N, and r is stated as follows: N = (r). By taking the logarithm of the two segments of the equation, the dimensions can be searched by the equation below:

$$Dim_{box}\ D = Lim_{r\rightarrow 0} \left\{ \frac{Log\ N_r}{Log\left(\frac{1}{r}\right)} \right\} \quad \ldots\ldots\ 2$$

Where log (Nr) is the number of boxes that cover the pore, log (r) is the measure of the pore length of the box's side.

3. Result and Discussion

Laboratory test results from the analysis of porosity, fractal pore dimension, and vitrinite reflectance ($R_{o,\ max}$,%) shows in table 2. The results show that four coal samples' porosity ranged from 2.38% to 2.63%, with an average value of 2.54%. The results of calculating fractal pore dimension using formula 2 of coal pores have values ranging between 1,933 - 1,964 (Table 2). Laboratory results from vitrinite reflectance analysis showed that the coal samples have $R_{o,\ max}$ ranging from 0.52% to 0.62% (bituminous)

Table 2. Results of porosity, permeability, fractal pore dimension, Vitrinite Reflectance, and Adsorption of methane.

Sampel code	Porosity, %	Permeability m Darcy	Fractal pore dimension, D	Vitrinite reflectan,%	Adsorption of methane, Scf/t
A	2.51	0.341	1.933	0.52	294
B	2.63	0.221	1.94	0.53	315
C	2.38	0.356	1.963	0.58	431
D	2.64	0.12	1.943	0.62	425

Each coal seam shows a different porosity value from seam A to seam D (Table 2). Coal seam C has a small porosity value of 2.38%, while coal seam A, B, and D, are 2.51% to 2.64%. Table 2 shows that the fractal pore dimension is inversely proportional to porosity, the higher the fractal pore dimension in the coal seam. C = 1,963, the porosity value is small = 2.38%. Coal seams A, B, and D have smaller fractal pore dimension than coal seam C, while the porosity value of coal seams A, B, and D are higher than seam coal C.

The higher the fractal pore dimension value, then the smaller the pore size. Whereas the fractal pore dimension also shows irregular pore distribution and the higher the fractal pore dimension, the more Irregular the pore distribution, and vice versa.

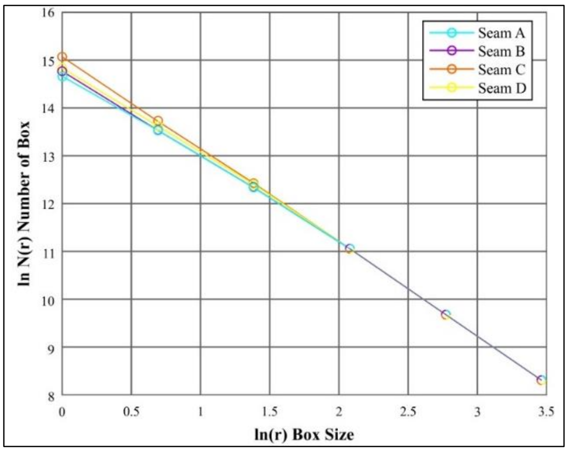

Fig 2. The results of data processing using the box-counting method

The pore fractal dimension is an intrinsic property of the pore surface of coal and coal structure related to coal rank and maceral composition (Nie et al., 2016). The characteristics of coal pores, including pore shape, pore distribution, and interconnected pores, determine the porosity and permeability of coal and affect gas uptake and transportation (C. J. Liu et al., 2015). Different pores will have different effects on the absorption ability of methane gas. Generally, micropores and mesopores are the main space for methane gas absorption. The pore surface area is inherently related to the pore size distribution, where the increasing pore surface area will cause a decrease in pore size for a given pore volume (Chalmers & Marc Bustin, 2007). The coal-burning process and the maceral composition have different effects on the pore surface and the inter-pore relationship, which will cause coal differences in gas absorption and permeability. During the coal process, the polycondensation of coal molecules will increase the coal rank (Fu et al., 2017). As a result of mass compaction during the polycondensation process, the rank of coal was increased, and the mesoporous and micropore pores were homogeneously distributed. The intensive polycondensation process of coal molecules will cause even mass compaction so that micropores and cracks will gradually develop in the coal (Fu et al., 2017); thus, coal will have a complex pore structure.

The ongoing coal-burning process will lead to the development of mesoporous and micropores abundant and evenly distributed. (X. Liu & Nie, 2016). This is evidenced by the increase in coal rank in the Tanjung Formation, which will increase the value of

the pore fractal dimension. The correlation of the pore fractal dimension with coal rank has determination coefficient (R^2) = 0.2367 (Figure 3).

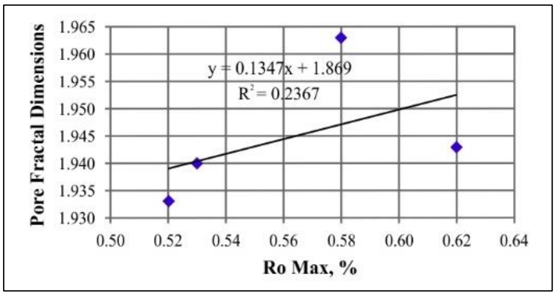

Fig 3. Relationship between coal rank and fractal pore dimension.

In general, the correlation between coal rank and fractal pore dimension shows a bad correlation. This correlation is bad because coal has almost the same value, Ro,max = 0.53% - 0.62%, which indicates a relatively similar value. Fractal pore dimension affects coal porosity; when fractal pore dimension increases in general, the porosity will decrease (Chen et al., 2015). The deeper the coal seam (Tanjung Formation coal) with Ro, maxs> 0.50% the more it has a slightly smaller porosity (Figure 4).

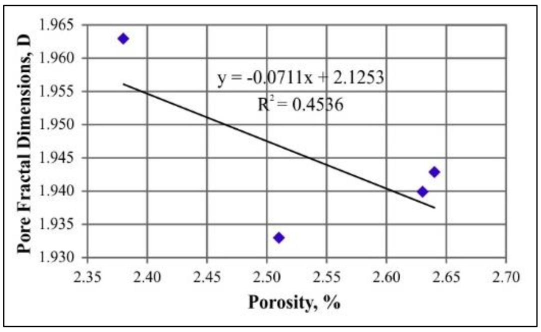

Fig 4. Correlation between Porosity and pore fractal dimensions with determination coefficient (R^2 = 0.4536)

The picture above shows that the greater the porosity value, the lower the fractal pore dimension, meaning that the lower the porosity, the more macropores and mesoporous numbers. The higher the fractal pore dimension, the greater the number of mesoporous and micropores, which will result in smaller porosity. Increasing the coal process and imperfect physical compaction will cause the coal pores, there are macropores, mesoporous, and fractures to be spread unevenly. As a result, it will cause the fractal pore dimension value to be smaller than higher rank coal. On the other hand, the coal-burning process continues and is combined with continued physical compaction. It will result in the pores in the coal, namely mesopores and micropores, which will be relatively evenly distributed on the coal, resulting in a large fractal pore dimension value.

Tanjung coal has a permeability between 0.12 - 0.356 m Darcy (Table 2). This data shows that the higher the rank of coal, the smaller its permeability. The permeability of Bituminous coal is relatively smaller because the pores that develop on the coal are mesoporous and micropores. In Bituminous coal, the pores are spread evenly and regularly on the coal surface due to the compacting; thus, it has relatively larger fractal pore dimension.

The absorption capacity of coal is highly dependent on the adsorption pores on its surface, generally micropores and mesopores (Chen et al., 2015). The size of the absorption of methane gas is also due to differences in depth (Anggayana, Kamarullah, Suryana, & Widayat, 2017). The deeper the seam of coal will cause, the greater the absorption of methane gas. Increasing the rank of coal will cause the pore volume in the micropore and mesoporous pores to decrease so that it will cause the porosity and permeability of the coal to be smaller. The absorption capacity also depends on the pore size distribution and the complexity of the pore surface structure (Chen et al., 2015). The smaller the pore size and the more complex the pore surface, the larger the pore surface area, which will result in greater absorption capacity of the methane gas. Increasing the rank of coal will increase the average diameter of micropores and mesopores, which will cause an increase in surface area (Chen et al., 2015). Increasing the rank of coal causes the pores of both micropores and mesopores to experience changes in their distribution; at the rank of Bituminous coal, the pores are evenly distributed (Figure 5).

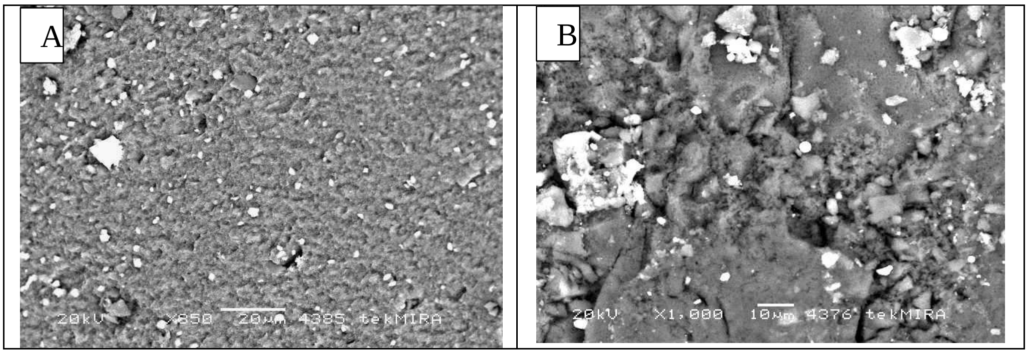

Fig 5. A. Photomicrography of SEM coal seam B shows regular pores. B. Photomicrography of SEM coal seam C shows irregular pores

The relationship between the fractal pore dimensions and the absorption volume of coal methane gas can be seen in Figure 6. Figure 6 shows that the correlation between methane gas absorption and fractal pore dimension has a positive correlation with determination coefficient (R^2) = 0.805. The larger the fractal pore dimension, the greater the gas absorption because coal has mesopores and micropores, which are spread evenly. Coal with small fractal pore dimension has a small absorption of methane gas, this is because the coal has macropores and mesopores that are spread unevenly. An increase in the rank of coal will increase methane gas absorption, which will increase the value of the fractal pore dimension.

According to (Yao et al., 2009) concluded that high pore structure is associated with pore heterogeneity, affecting gas adsorption. However, from the research results, researchers where the fractal pore dimension have a positive correlation with adosprtion of methane. When fractal pore dimension increase in small pores increase compared to large pores in coal, the effect of small pores on in surface area is much higher than in large pores. As a result of the small pores that develop, the fractal pore dimension value becomes large; this is shown in the coal seam C, which has large fractals.

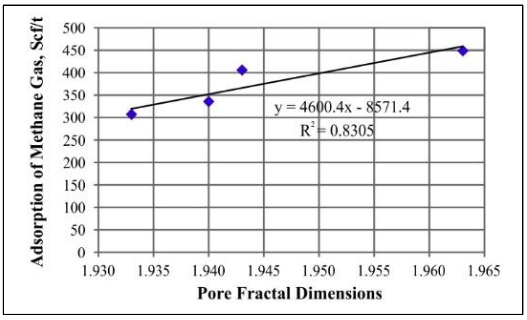

Fig 6. Relationship between adsorption of methane gas and Fractal pore dimension

Based on previous research by (Li et al., 2015) , micropores have a large surface area and can provide a large absorption of methane gas. Gas absorption does not depend on pore volume but depends on the pore surface, and ordinary pore surface will have less methane gas absorption than the irregular pore surface. Meanwhile fractal pore dimension are related to micropores, where a decrease in pores causes a more regular pore diameter, which results in a decrease in fractal pore dimension.

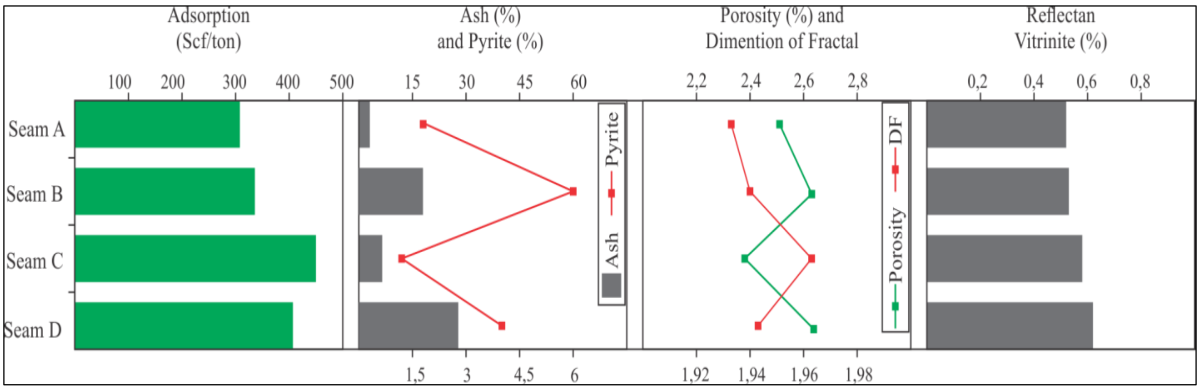

Fig 7. The Value of adsorption methane gas, Ash, Reflectance Vitrinite, and Pyrite content

In seam C it is known that the largest absorption of methane gas, while the fractal pore dimension value is large, shows that the coal seam shows many micropores and a relatively rough surface. The fractal value is influenced by whether the pore surface is regular or not. If the pore surface is regular, the pore surface will cause a small absorption of methane gas, but if the pore surface is irregular, it will cause a large methane gas absorption. The coal seams A, B, and D have small fractal pore dimension value compared to coal seam C, and this shows that coal seams A, B, and D have a regular porous surface while coal seam C has an irregular porous surface. The pores surface shows that the absorption capacity of coal seams A, B, and D is smaller than seamless coal C, see Figure 7.

Ash and pyrite minerals are impurities in the coal, especially on pore surfaces, which will affect the absorption of methane gas (Figure 7). Previous researchers (Laxminarayana & Crosdale, 1999) explained that ash content and the mineral matter would reduce methane gas's absorption capacity. Besides that, mineral matter, especially pyrite, will fill micropores, which will reduce the absorption capacity of methane gas. Minerals on the pore surface influence the regularity of the micropore surface the presence of pyrite minerals that fill inertinite group macerals, especially fusinite, semifusinite, and sclerotinite (Figure 8).

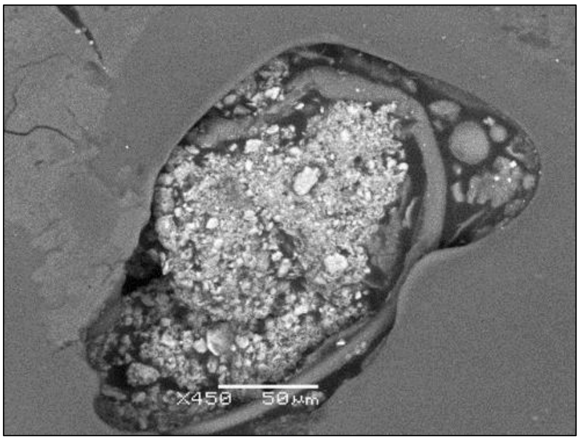

Fig 8. Photomicrography of SEM shows pyrite minerals that fill inertinite group maceral

If the pyrite mineral meets the maceral group, it will cause irregularity. The microsurface, which causes the fractal pore dimension to be small, will cause the absorption of methane gas to decrease. In comparison, coal rank has an important influence on the fractal pore dimension. However, coals of the same rank will have almost the same fractal pore dimension. According to the research results by (Yao et al., 2009) on carbon content, the more coal that contains relatively high carbon, the higher the fractal pore dimension of coal, which have a relatively similar rank of coal. The coal seam C has a higher fractal pore dimension and adsorption of methane gas than the coal seams A, B, and D, which shows that coal seam C has a higher carbon content than coal seams A, B, and D.

4. Conclusion

The fractal pore dimension calculation using the Box counting method can determine whether the pore volume and pore surface are regular or irregular. Coal with irregular pores has a large fractal pore dimension, while regular surface pores have small fractal pore dimension. The irregular surface of the pores affects the absorption of methane gas. Coal with a regular pore surface will absorb less methane gas than an irregular pore surface. The absorption of methane gas increases with the rank of coal. In coal, which has almost the same rank, the fractal pore dimension difference is minimal. Seam C coal has abundant methane gas absorption with large fractal pore dimension, but its porosity is small because this pore volume automatically affects the absorption of methane gas. The value fractal pore dimension of seam C is high, indicating that coal has a surface and pore structure heterogeneity. Fractal pore dimension can be used to determine the size of coal porosity.

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