

The Effects of the Petrophysical Properties of Calcareous Mudstones and Limestones on Capillary Pressure Values*

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Search and Discovery Article #41453 (2014)

Posted September 22, 2014

*Adapted from extended abstract prepared in conjunction with poster presentation at AAPG International Conference & Exhibition, Istanbul, Turkey, September 14-17, 2014, AAPG©2014

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Abstract

Fifteen calcareous mudstone and limestone samples were selected from over one hundred and twenty sandstone, limestone and siltstone samples from the Oklahoma Panhandle. These samples were analyzed using the Auto Pore IV Mercury Intrusion Porosimeter, manufactured by Micromeritics, to examine their petrophysical properties of capillary pressure (psi), pore radius (μm), mean radius (μm), cumulative pore volume (mL/g), incremental pore volume (mL/g) and percentage of total Hg intrusion volume. In this research, mudstone and limestone porosities were measured and then the porosity effects on capillary pressure P_c and displacement pressure, at 10% of mercury intrusion P_d were analyzed. Porosity is important especially in the petroleum industry when differentiating between different sediments and the purpose or role in which it serves (differentiating between reservoir or cap rocks). From this analysis both the P_c and P_d , values were obtained graphically.

Introduction

The current analysis of these mudstone and limestone samples serves also a larger purpose as it helps in the interpretation and prediction of the height of carbon dioxide (CO_2) retention in the already depleted gas fields of the Oklahoma Panhandle ([Figure 1](#)). The results of this study will be used to examine what role and how important porosity is in the carbon dioxide sequestration processes. It would highlight the retention capabilities of the calcareous mudstone and limestone samples and how effective they may act as seals in preventing the leakage of CO_2 to adjacent layers when stored. Carbon dioxide and other greenhouse gases have been responsible for the earth-atmospheric energy imbalance and have caused great concern to the future of the earth's

atmosphere. To eliminate the presence of CO₂ in the atmosphere would assist in restoring the energy balance of the earth and may alleviate the current warming trend of the earth's surface.

Methods

Fifteen calcareous mudstone and limestone samples were broken into smaller pieces. They were then heated using an electric oven for 24 hours to ensure that all moisture was eliminated and the pores were free from any fluid. The samples were then weighed and the weight was recorded. Next, the samples were loaded into a penetrometer and then reweighed and the weight once again recorded.

To begin the analysis the penetrometer was loaded into the low-pressure port of the Auto Pore IV a petrophysical device. A sample information file was created using the Auto Pore IV software, where all information of the sample, analysis conditions and all other parameters were recorded. The low-pressure analysis began with gas being evacuated from the penetrometer by vacuuming and mercury immediately filling the penetrometer. This first phase of analysis ended with the collection of low-pressure data (0 - 50 psi). Once this data was collected the penetrometer was removed from the low-pressure port and weighed, its weight recorded and then it was transferred to the high-pressure port.

Analysis in the high-pressure port measured and collected pressure data up to 60,000 psi. This analysis also ended with the collection of high pressure data ranging from the first of the high pressure points measured to the last. The data from both the low-pressure and high-pressure ports were collected in an Excel format. The values recorded were the pressure (psi), mean radius (μm) and total Hg intrusion volume (%). These values were further analyzed using the OriginPro 7 software.

Study Area

The Oklahoma Panhandle is located to the extreme west of Oklahoma State and consists of three counties: Cimarron, Texas, and Beaver Counties ([Figure 1](#)). Sediments in this area are considered to be deposited during the Late Mississippian and Early Pennsylvanian time period continuing until Permian. They belong mainly to two formations, the Springer and the Morrow. The Springer formation was deposited in the Late Mississippian and is composed primarily of sandstone, shale and limestone. The paleoenvironment during deposition was open marine and this accounts for its sedimentological composition.

The Morrow Formation was deposited during the Early Pennsylvanian and is comprised of mainly coarse-grained sandstones and conglomerates. Like the Springer, most Morrow sediments were derived from fluvial corridors, therefore resulting in intermingled sandstone facies of fluvial, tidal and marine deposits that eventually shale out (Andrews, 2008). Unlike the Springer formation, the Marrow Formation is divided into three sections: the upper, which is composed of marine shale, the middle, composed of limestone called the Union Belly Limestone, and the lower Morrow, composed entirely of sandstone. There is a distinct difference between the shales found in the Springer and Morrow Formations. The Springer shales tend to be dark in color and less calcareous whereas the shales found in the Morrow Formation are calcareous and light in color.

Discussion

Porosity is the ratio of void spaces in a rock sample relative to its total volume. A collection of pore spaces and pore throats combined can be referred to as a pore system. This is crucial in understanding reservoir and sealing capacity characteristics. Pore spaces can be classified in four pore geometries: intergranular, intercrystalline, vuggy/moldic and fracture. These pore geometries are then further classified into two pore system groups: petrophysically simple Archie porosity and petrophysically complex Non-Archie porosity. Pore system shapes and their characteristics using Archie and Non-Archie porosity are shown in [Table 1](#) (Choquette and Pray, 1970; Hartman et al., 1999).

The capillary pressure or P_c ([Figure 2](#)) value is the difference in pressure between two immiscible fluids across a curved interface at equilibrium (Tiab et al., 2004). Capillary pressure curves are rock property measurements that relates the volume of pore space controlled by pore throats of a given size (microns) to a given capillary pressure (Hartman et al., 1999). This is important since it gives: 1. an estimate of the pressure required to enter the smallest pore spaces or pore throats of a rock, 2. the size of the pore throats, and 3. the water-hydrocarbon saturation at different pressures. Capillary pressure is expressed as:

$$P_c = \frac{2\gamma \cos \theta}{r} \quad (1)$$

where P_c is the capillary pressure, γ is the surface tension of the liquid, θ is the wetting angle of the liquid and r is the effective radius of the interface (Hartman et al., 1999).

The displacement or breakthrough pressure or P_d value ([Figure 2](#)) is the minimum pressure required to begin saturating the rock sample with a nonwetting phase (mercury) (Cranganu, 2011). The magnitude of the breakthrough pressure is determined by the highest capillary pressure of an interconnected network of pore throats that are first invaded by the nonwetting phase (Li et al.,

2005). P_d is the pressure needed for mercury to fill 10% of rock pores (Schowalter, 1979; Cranganu, 2011). Bulk density is defined as the weight per unit volume of material. It is important in petrophysical measurements since it gives an idea of the rate at which moisture moves through a material, the formation and accumulation of sediments such as clays and carbonates.

Results

In the [Table 2](#), three columns were analyzed and compared to see if there were any trends, changes and connections amongst them. The three columns compared were the P_c , P_d and porosity ϕ columns. The P_c column was first analyzed and it was seen that it consisted of both low and high-pressure values ranging from 3.1×10^0 psi to 1.1×10^4 psi. The sample that required the lowest pressure for infiltration was sample 1461 and its lithology was a white fine grain limestone whilst the sample that required the highest pressure for infiltration was sample 2472 and its lithology was a light grey fine-grained mudstone.

Next, the P_d column was analyzed and unlike the P_c column, it contained low-pressure values with the exception of two samples 878 and 3138 that had high-pressure values of 1.7×10^3 psi and 9.1×10^2 psi respectively. The lithology of sample 878 was a black fine grain calcareous mudstone whilst the lithology of sample 3138 was a light grey medium grain sandy limestone. Even though these pressure values were the higher-pressure values in the P_d column, they were still relatively low compared to the pressure values found in the P_c column. Sample 1822 had the lowest pressure value of 0.7×10^{-1} psi and its lithology was a black fine grain calcareous mudstone. Lastly, the porosity column was analyzed. It too contained a combination of low and high percentages ranging as low as 2 % to 27%. Lower percentages however, were more dominant in the samples than higher percentages. The sample with the lowest percent porosity was 3146 and its lithology was a red fine-grained calcareous mudstone whilst the sample with the highest porosity was 3952 and it was black fine grain fissile calcareous mudstone.

In this study, fifteen samples of two rock types were studied and analyzed; they were twelve calcareous mudstones and three limestones. For both rock types, their grain size ranged from fine to medium to coarse grains, and their colors, pink to purple, grey to black. The calcareous mudstones samples exhibited relatively high P_c pressure values with the exception of samples 1081 and 1822 that exhibited low-pressure values ([Table 2](#)). P_c values are a representation of the small pores within a rock and are usually the last pores to be filled with a nonwetting fluid. The calcareous mudstone samples consisted mainly of fine grains and this could account for the relatively high P_c pressure values for infiltration of the nonwetting fluid. Since these samples consisted of fine grains, this means that they were compacted and well sorted which can also account for their high P_c values.

Samples 1081 and 1822 were also finely grained, however, instead of having high-pressure values they displayed relatively low-pressure values. The cause for this could be that they were poorly sorted and as a result having more open spaces between them for infiltration to take place. The P_c pressure values for the three limestone samples consisted of both high and low pressure values ([Table 2](#)). One sample in particular 1461 displayed an unusual low P_c value. A reasonable explanation for this is that when limestones come into contact with moisture the calcium carbonates dissolve and its pore structure becomes deformed forming karsts. Karsts are holes made when calcium carbonates dissolve. This could explain the relatively low P_c value in sample 1461 since more and larger pores were available for the infiltration of fluid.

P_d pressure values are a representation of large pores within a rock and are usually the first pores to be filled with a nonwetting fluid. All the samples displayed low P_d values except one sample 878 ([Table 2](#)) that displayed a high P_d value. Since its grain size was fine grained, the possibility is that it was extremely compacted and as a result required high pressures for infiltration to take place. Porosity consisted of both high and low percentages ([Table 2](#)), porosity, however, relied on the amount of pore spaces within the rock sample. In relation to P_d and porosity, there was no trend; however, P_c did have an effect on porosity. It was shown that the higher the P_c values the lower the porosity, and the lower the P_c value the higher porosity ([Table 2](#)). Sealing capacity is the maximum pressure, which the smallest pore spaces of a rock, is filled with a nonwetting fluid. This is where the P_c line in [Figure 2](#) below is shown and is important when selecting a rock type to act as a seal since this gives an idea of the maximum pressure needed to break a rock. In a situation where the plateau is not well defined due to instances of poor sorting as shown in [Figure 3](#), a P_c line would be unable to be drawn; as a result, the P_d line, which is the displacement pressure, at 10% of mercury intrusion, can be used. The P_d can be used in this case since it gives the pressure needed to fill the larger pores and as a result would give a rough estimation of the pressures needed to fill the smaller pores in the rock sample.

Lastly from analyzing the porosity (%) versus P_c (psi) and porosity (%) versus P_d (psi) value graphs ([Figure 4](#) and [Figure 5](#)) it can be seen that from 0-10% porosity the P_c values are high as compared to the P_d values from 0-10% which are totally opposite, low. These graphs prove that between 0-10% porosity, very high pressures are needed to infiltrate the smaller pores in a rock sample whilst very low pressures are needed to infiltrate the larger pores in a rock sample. This is a key characteristic in the petrophysical properties on capillary pressures since this gives an idea of what rocks may make good seals. The higher the P_c and P_d in conjunction with low porosities would have a better sealing capacity. The more well sorted a sample the lower its porosity.

Conclusions

Fifteen calcareous mudstone and limestone samples were selected from the Oklahoma Panhandle and analyzed where its petrophysical property of capillary pressure (psi) was analyzed. From this research, the mudstone and limestone porosities were measured and then the porosity effects on capillary pressure P_c and displacement pressure, at 10% of mercury intrusion P_d were examined. The results of this study showed that smaller pores in a rock sample usually require a high capillary pressure for infiltration of a nonwetting fluid to penetrate into the rock whilst the larger pores require a small capillary pressure. At 0-10 % porosity, the P_c values are high as compared to the P_d values from 0-10%, which are low. This is key evidence proving that in the initial stages of infiltration very high pressures are needed to infiltrate the smaller pores in a rock sample whilst very low pressures are needed to infiltrate the larger pores in a rock sample. This characteristic is important since this gives an idea of what rocks may make good seals aiding in the elimination of carbon dioxide in the atmosphere. Rocks that are well sorted and contain both high P_c and P_d values with a low porosity would tend to have a greater sealing capacity as compared to rocks that do not have high P_c and P_d values. From analyzing the fifteen rock samples, one sample had the characteristics necessary for a good sealing capacity, sample 878. It had a P_c value of 6359, a P_d value of 1781, and a porosity value of 6.6%. Its lithology was a black fine grain calcareous mudstone. Rocks with characteristics like this would definitely have a good sealing capacity and would make good seals and assist in the bigger project of the interpretation and prediction of the height of carbon dioxide (CO_2) retention in the already depleted gas fields of the Oklahoma Panhandle.

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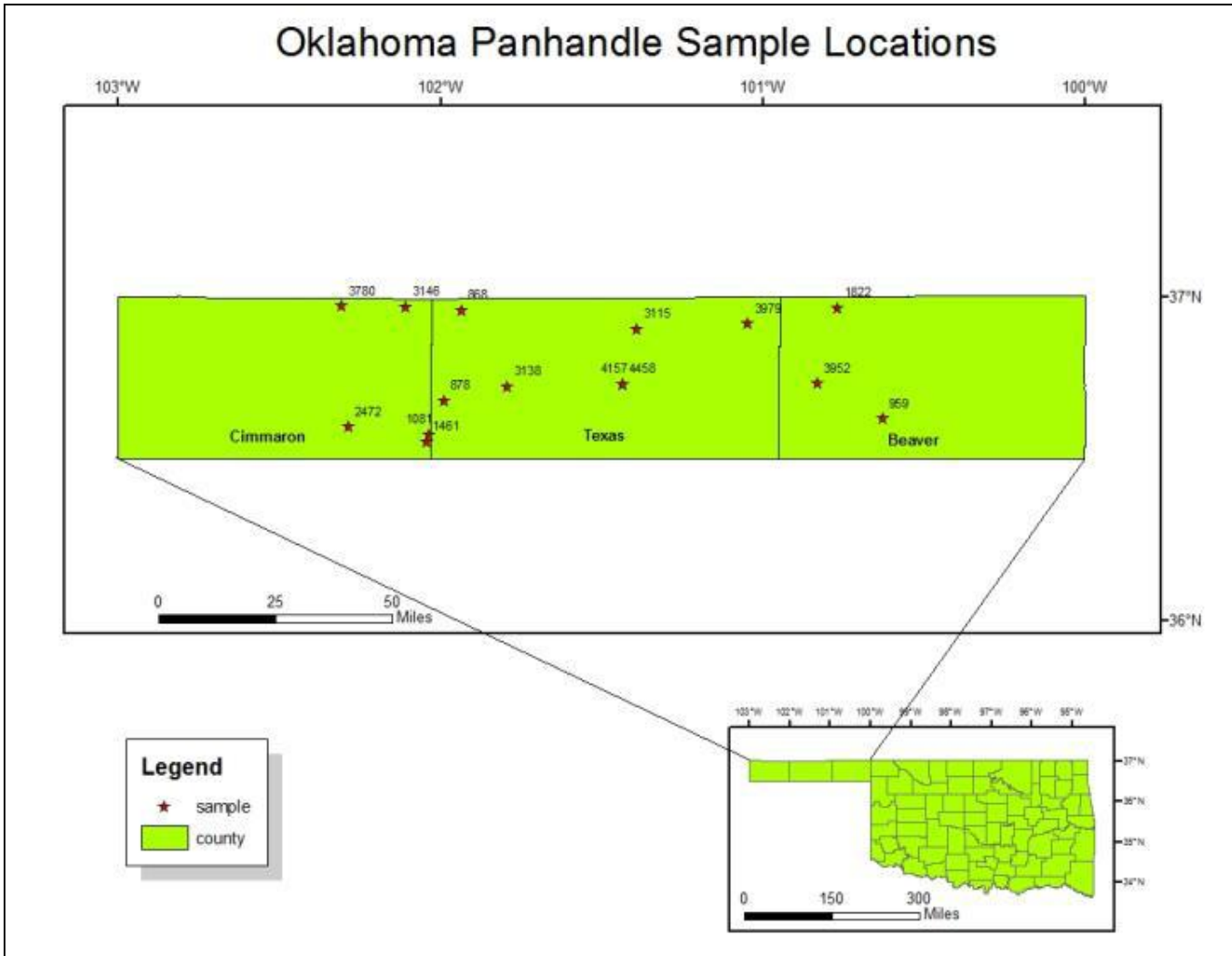


Figure 1. Oklahoma Panhandle geology sample locations.

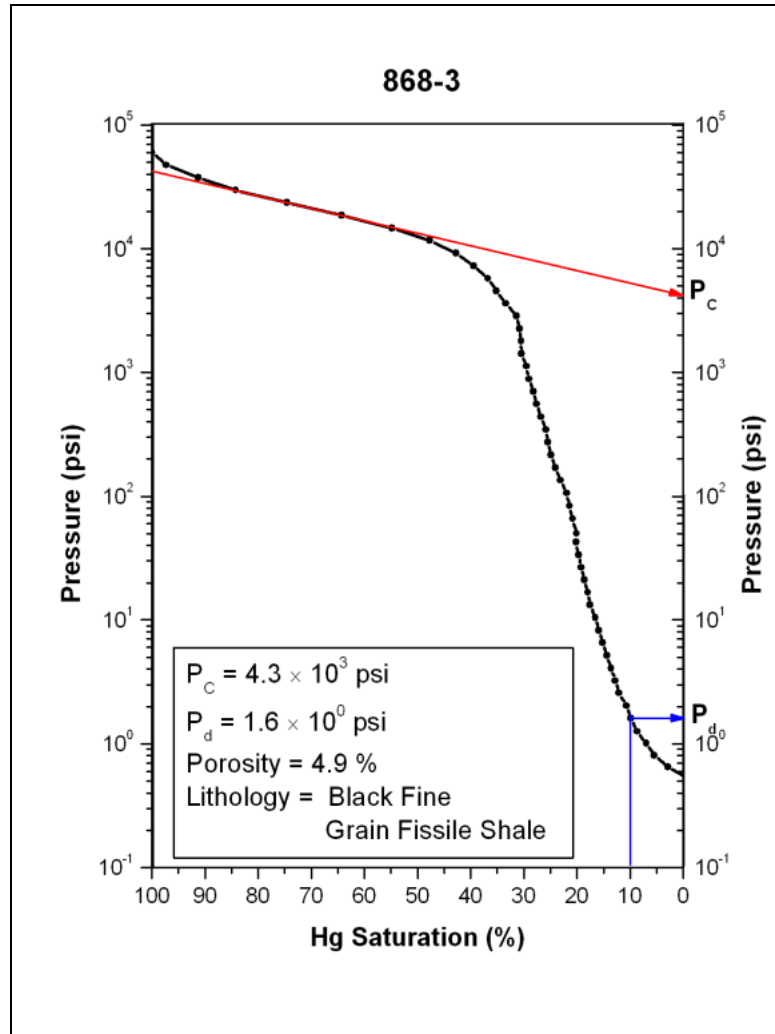


Figure 2. P_c and P_d pressure values.

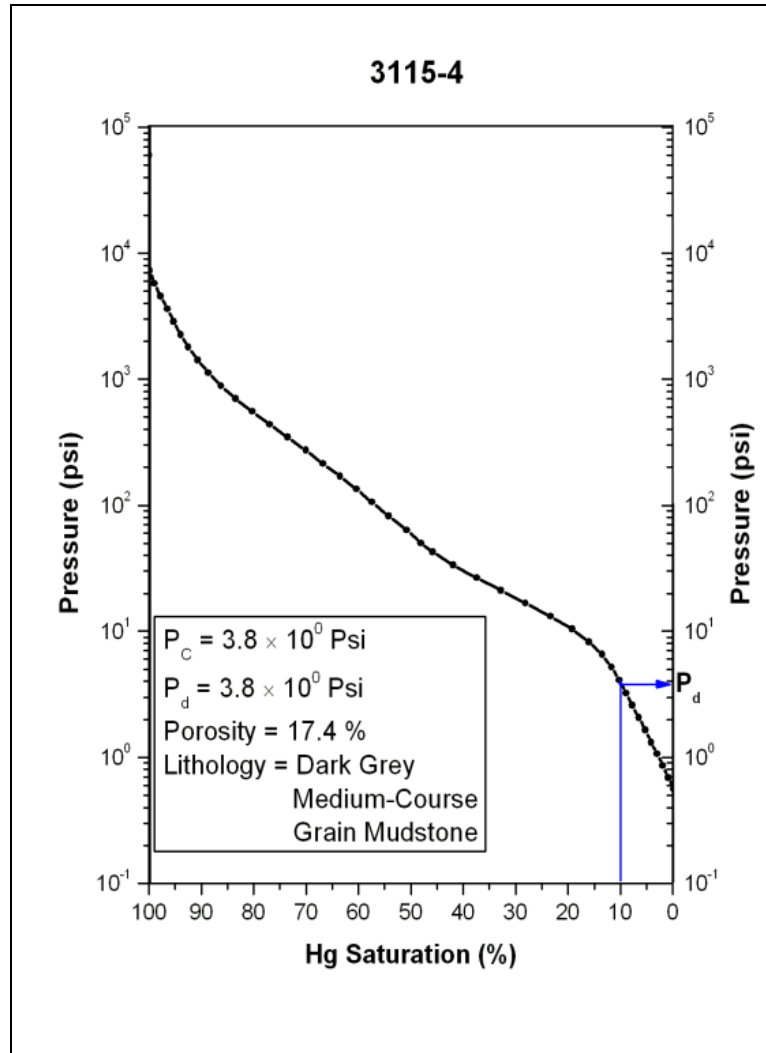


Figure 3. An undefined plateau.

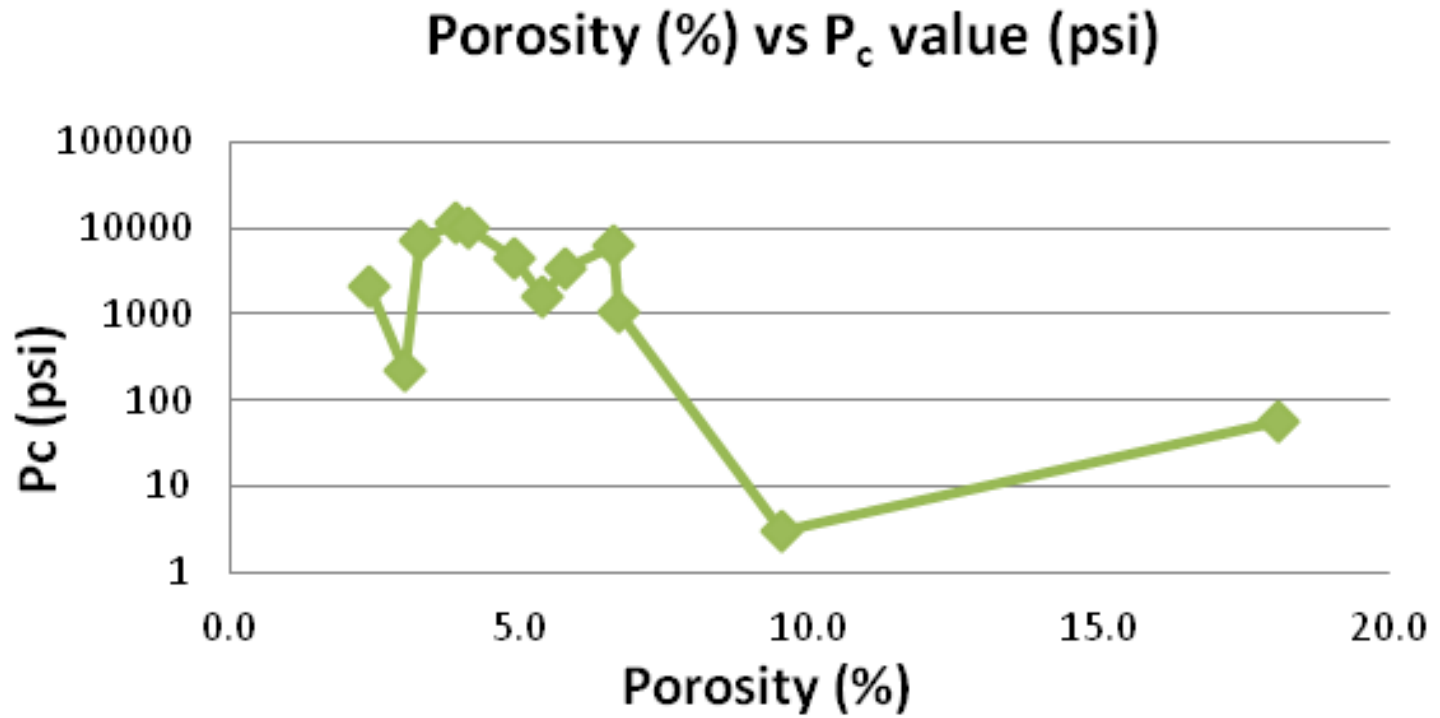


Figure 4. Porosity verses P_c value graph.

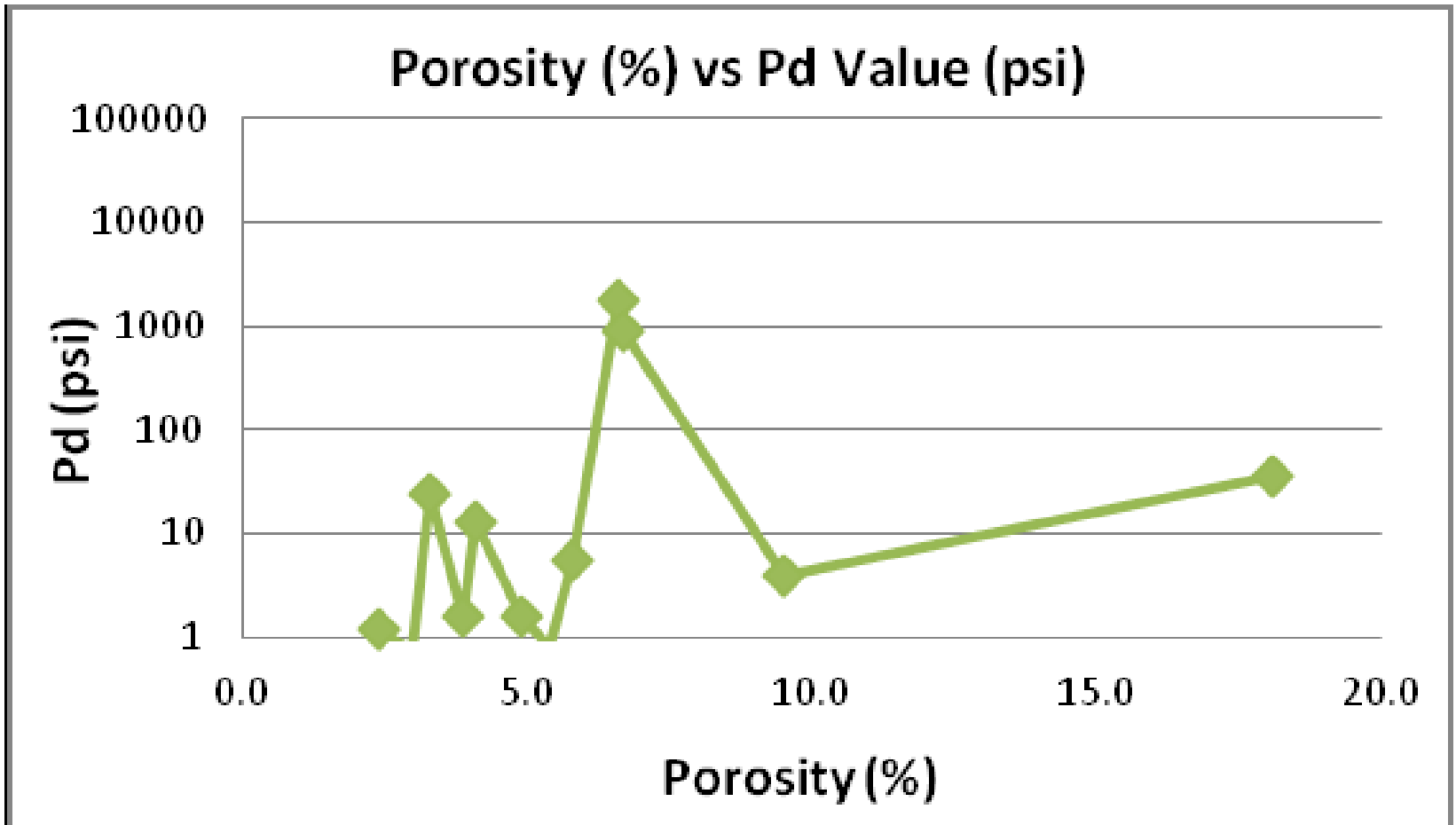


Figure 5. Porosity verses P_d value graph.

Feature	Archie	Non-Archie
Pore system shapes	Intergranular (found between rounded particles); Interparticle Intercrystalline (found between angular particles)	Mold-like Intraparticle Moldic Shelter
		Vug-like
		Boring/burrow Growth- Framework Fenestral Vug/channel/cavern
		Fracture-like Fracture Shrinkage
Pore Connectivity	Pore throats connect pores into regular networks	Pores are irregularly distributed and can be either poorly or very well connected

Table 1. Characteristics of pore shapes and their relationships to Archie and Non-Archie Porosity (Choquette and Pray, 1970; Hartman et al., 1999).

Sample #	Longitude	Latitude	County	Formation/Age	Lithologic Description	Top (ft)	Bottom (ft)	P _c (psi)	P _d (psi)	φ (%)	Bulk density (g/cm ³)
868	-101.93526	36.95927	TEXAS	Purdy/ Pennsylvanian	Black Fine Grain Fissile Shale	4524	4547	4318	1.6	4.9	2.49
878	-101.98941	36.6806	TEXAS	Cherokee/ Pennsylvanian	Black Fine Grain Calcareous Mudstone	4524	4600	6359	1784	6.6	2.41
959	-100.62811	36.62549	BEAVER	Council Grove/ Upper Permian	White Medium Grain Limestone	3611	3692	N/A	21	9.8	2.39
1081	-102.03385	36.57557	CIMARRON	Topeka/ Pennsylvanian	Pink Fine Grain Mudstone	3462	3573	56	35	18.1	2.28
1822	-100.76728	36.9666	BEAVER	Oswego/Upper Ordovician	Black Fine Grain Calcareous Mudstone	5630	5690	220	0.7	3.0	2.59
2472	-102.28673	36.59978	CIMARRON	Topeka/ Pennsylvanian	Light Grey Fine Grain Mudstone	3538	3566	11144	1.6	3.9	2.55
3115	-101.38974	36.90123	TEXAS	Keyes/ Pennsylvanian	Dark Grey Medium-Course Grain Mudstone	6533	6556	N/A	3.8	17.4	2.21
3138	-101.79176	36.72436	TEXAS	Marmaton/ Pennsylvanian	Light Grey Medium Grain Sandy Limestone	5855	5902	1059	913	6.7	2.45
3146	-102.10679	36.97092	CIMARRON	Keyes/ Pennsylvanian	Red Fine Grain Calcareous Mudstone	4540	4684	2112	1.2	2.4	2.61
3780	-102.30638	36.97337	CIMARRON	Cherokee/ Pennsylvanian	Dark Grey Fine Grain Calcareous Mudstone	3972	4003	7093	24	3.3	2.50
3952 (?)	-100.83059	36.73403	BEAVER	Chester/ Mississippian	Black Fine Grain Fissile Calcareous Mudstone	6770	6794	N/A	1.6 (?)	26.9 (?)	1.94
3979	-101.04653	36.91751	TEXAS	Atoka/ Pennsylvanian	Black Fine Grain Calcareous Shale	6450	6486	3488	5.4	5.8	2.47
4157	-101.43583	36.73106	TEXAS	Morrowan/ Pennsylvanian	Purple Fine Grain Mudstone	5985	6012	10242	13.1	4.1	2.54
4458	-101.43583	36.73106	TEXAS	Morrowan/ Pennsylvanian	Pink-white Calcareous Mudstone	5985	6012	1631	0.8	5.4	2.50
1461	-102.04292	36.55374	CIMARRON	Topeka/ Pennsylvanian	White Fine Grain Limestone	3455	3565	3.1	3.9	9.5	2.42

Table 2. Information and analytical results (P_c, P_d, φ) of the fifteen samples.