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Methods Improve Shale Core Analysis

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HOUSTON–The fundamental properties of gas shales make them virtually impossible to analyze with conventional core analysis methods or petrophysical models based on log data. This includes permeabilities in the tens to hundreds of nanoDarcies, low effective porosities (typically less than 10 percent), and high kerogen and clay contents. The tightness of the rock and the abundance of clay minerals and kerogen generally creates a number of technical challenges to core analysis.

Permeability, effective porosity, and oil, water and gas saturations are fundamental reservoir properties for assessing in-place hydrocarbons, producibility and overall economics. In unconventional shale systems, porosities are very low and permeabilities are 100 to 1,000 times lower than what was considered "tight" only a few years ago. Since a fraction of a small number is even smaller, it follows that the oil, water and gas content in tight shale pore space is also very small.

However, the abundance and volumetric extent of these plays are large. Some two-thirds of the sedimentary rocks in the earth's crust are shales. Their lack of significant porosity and pore-filling hydrocarbons is amply compensated by volumetric extent, providing favorable conditions for economic production. The problem is how to evaluate the properties of these tight, small-particle sized systems.

Analysis of the microstructure of tight shales suggests four types of porosity, with organic and nonorganic porosity being the dominant differentiators. Organic porosity may be the most relevant porosity for hydrocarbon accumulation and production. The microstructure also suggests a biased distribution of fluids, with hydrocarbons predominantly hosted in the organic porosity and brines in nonorganic pores. The separation and potential hydraulic discontinuity between brines and hydrocarbons pose important questions to standard concepts of pore saturations and the relative mobility of fluids in pore space.

In addition, a larger portion of the water content in tight shales is immobile, either through capillary, double-layer, or structural forces of varying magnitudes. Defining and discriminating "free" pore water from "bounded" or "matrix" water is a challenge.

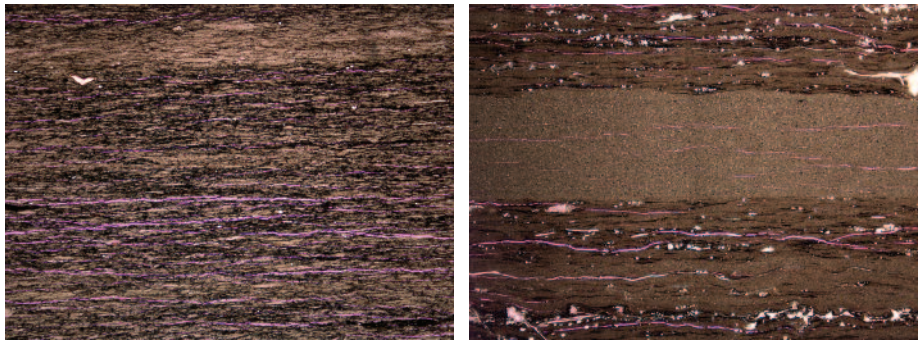
To understand reservoir properties in tight shales, it is necessary to not only reconsider the meaning of previously defined concepts for conventional porous media, but also to find a new way to measure them. New analytical methods have been developed to evaluate cores from gas shale reservoirs by using crushed material to enable better access to the pore space, a high-throughput retort system practical for commercial-scale analysis to separately measure free/bound/structural water volumes, visually distinguish water from oil, and pressure transient analyses to determine permeability.

Applying conventional core analyses to kerogen- and clay-rich rocks fails to separate free from bound waters, and water from light oils, thereby missing critical inputs into calculating effective saturations, effective porosities and clay-bound water volume. In addition, the amount of oil recovered from retort, as an independent quantity, can be a useful proxy for kerogen maturity. In conventional reservoirs with large pore volumes and low clay content, these distinctions are more muted.

In regard to permeability measurements, unconventional reservoirs are usually too tight to allow for steady-state methods, and microfracturing is often too pervasive to allow for reliable permeability measurements on whole plug samples. Crushed sample pressure decay

FIGURE 1

Microfracturing in Gas Shale Thin Section Images



systems can measure the nanoDarcy permeabilities typical of shales, while a stepped confinement pulse-decay method can be used to measure microDarcy (and higher) permeabilities in more texturally complex, siltier or sandier unconventional reservoirs with higher permeabilities, where crushing the sample would affect the results.

Understanding The Rock

Understanding the rock (including composition, texture and variability at small and large scales) is fundamental to reservoir characterization and laboratory measurements in tight shales. Particular aspects of unconventional rocks that make conducting these measurements complex are the colloidal size of the sediments, their planar contacts, the mixture of organic and inorganic components, and a ubiquitous capacity for remineralization.

Mechanically, the planar contacts result in low stress concentrations at the grain-to-grain contacts, and low propensity for grain crushing and changes in rock structure. This is in contrast to sandstones and siltstones, which exhibit more localized contacts, develop high stress concentrations at the grain-to-grain boundaries, and have a high propensity for changing rock structure with stress.

Conversely, in tight shales, the presence of organic matter provides soft inclusions for local stress redistribution, and the contacts between the organic and inorganic matter are weak and prone to tensile and shear failure. The colloidal size of tight shale sediments results in high surface area per volume ratio, and in a high corresponding surface energy. This is subsequently minimized through bio-geochemical interactions between the organic and inorganic constituents and often strongly catalyzed by interactions with living organisms. The combined effect results in high diagenetic transformations and continuous remineralization of the system.

Core observations show that fractures and partings in tight shales get fully mineralized, whether hairline or significantly wide fractures. Observations on thousands of feet of core show a pervasive presence of subvertical mineralized fractures (veins) on most vertical cores, with few exceptions. In some cases, such as the Haynesville Shale, the mineralized veins are oriented subhorizontally, suggesting the presence of veins is pervasive and represents an important property of these systems.

The result is a rock fabric containing the overprint of the original depositional system, a microstructure substantially transformed by diagenetic processes, and large sets of veins with subvertical to subhorizontal orientations. This complex fabric results in complex distributions of properties (heterogeneity). The preferential fabric orientation results in material properties that also are preferentially oriented, and this anisotropy is not only limited to their elastic behavior, but also impact strength, porosity, flow potential, thermal conductivity and all other properties.

Core Sample 'Microcracking'

One consequence of the complex rock fabric and weak organic/inorganic contacts in tight shales is that a large number of partings are induced by stress unloading and core relaxation during coring and core retrieval.

The resulting "microcracking" increases porosity, and more importantly, significantly increases permeability in tight shales, providing unrealistically high permeabilities and inconsistent results. Figure 1 shows thin section images of a tight gas shale in which microfracturing is denoted by pervasive pink streaks, caused by infusing the sample with a pink epoxy during preparation. Understanding the effect of these artifacts on rock properties, and their consistent removal prior to initiating property measurements, is the dominant focus in laboratory testing of tight shales.

The most consistent method for eliminating microcracking and reliably assessing matrix porosity and permeability is crushing the samples and sieving the fragments to a size that is representative of the rock microstructure. Anisotropic materials subjected to stress deform more in a direction perpendicular to bedding rather than parallel to bedding, which means they may be subject to a considerable amount of shear strain. This results in shear displacement of the induced microcracks as they are created, making them more difficult to close by reloading,

FIGURE 2

SEM Image of Crushed Rock Sample for Retort Analysis

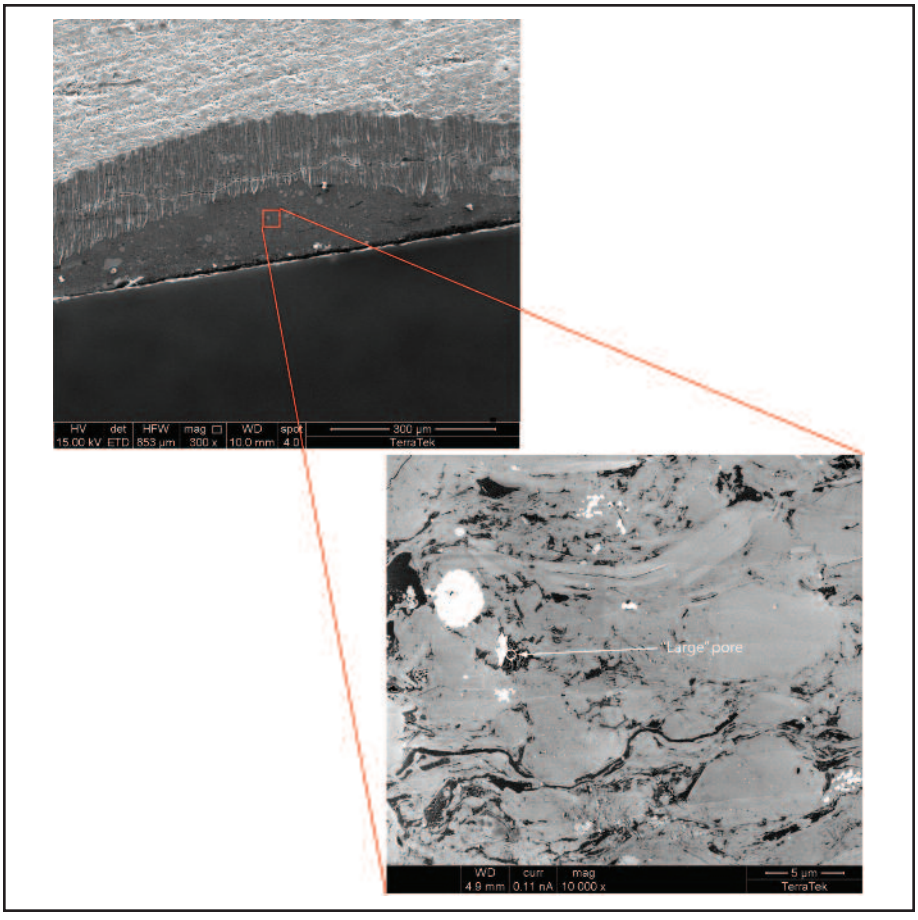
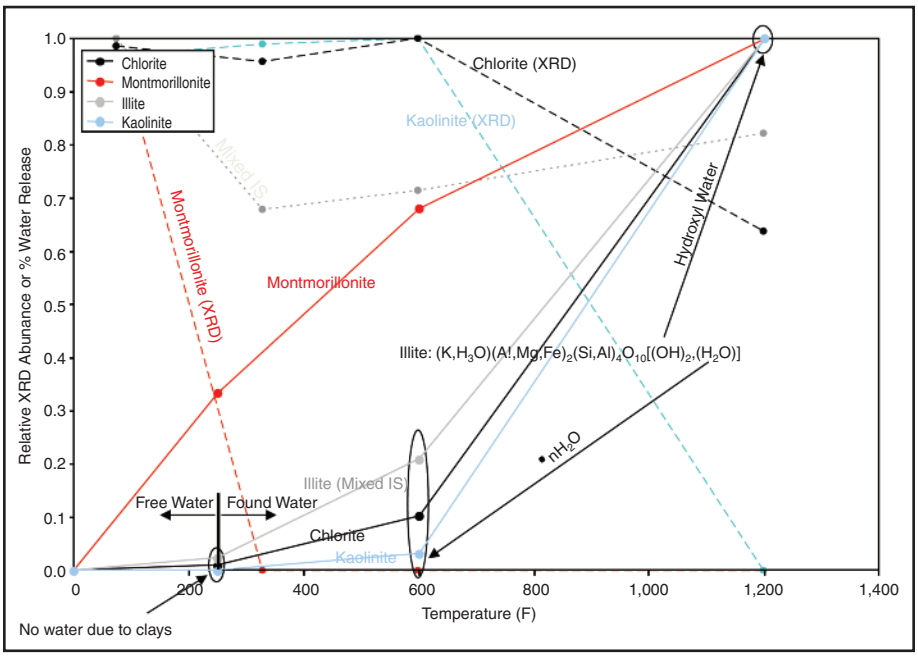


FIGURE 3

Three-Step Retort of Clay Standards and XRD Mineral Analysis after Each Step



in contrast to isotropic systems. While most microcracks are minimized or eliminated with increasing stress in some rocks, in others, remnant microcracks remain open even at stresses higher than the mean stress.

Using rock crushing for retort extraction and permeability measurements must include a strong understanding of the final size of the crushed material in relation to the rock microstructure. The goals are to eliminate coring-induced artifacts while maintaining a representation of the pore structure within the crushed material. The crushed rock fragment size needs to be sufficiently small to remove all microfractures, yet large enough to be representative of the rock matrix.

Since pore sizes in tight gas formations tend to be up to tens of microns in size at the upper end, crushed fragments in the millimeter-size range contain hundreds of thousands of pores, and can be considered representative of the overall matrix structure. Figure 2 shows a scanning electron microscope image of a crushed rock fragment used for retort analysis. The zoom-in shows a portion of the sample with a larger-than-average pore highlighted.

The added benefit of rock crushing is that it increases the surface area-to-volume ratio of the samples, greatly decreasing the time needed for fluid extraction, pressure diffusion and transient-state permeability measurements.

However, there are situations where rock crushing may be detrimental to the analysis, such as when the rock has an organic-rich, low clay maturity, poorly cemented and deformable matrix. In this case, the crushing process may exceed the yield strength of the rock and induce considerable plastic deformation, changing the pore structure. For thinly laminated rocks (laminated silty or silty/sandy mudstones) to tight gas sands with high detrital content, the crushing process may eliminate textural features by removing the interfaces between high-permeability and low-permeability layers.

Furthermore, increasing the surface area by creating small fragments increases the propensity of capillary suction, capillary condensation and drying. Therefore, proper care should be exercised so that the native saturation state is not altered. Finally, rock crushing may eliminate real microcracking present in situ, and while providing a good assessment of the matrix properties, could underestimate porosity and permeability. This may be the case in shallow reservoirs that have been uplifted by isostatic rebound (the Antrim Shale, for example).

Core Handling

It is important to understand that all laboratory testing is conducted on samples under an "as received" state, meaning they may not be in their unaltered "native state" prior to core retrieval. Even when subjecting core samples to simulated in situ conditions prior to testing, the fact that the rock has experienced ambient conditions means this transition created changes in porosity (microcracking), permeability (microcracking and fluid invasion) and saturations (fluid invasion, phase changes, liquid expulsion). While the very low permeability of shales minimizes some of these effects, unfortunately, changes from the native state are difficult to reconcile and can be exacerbated by improper core handling.

In addition, the intergranular porosity can be predominantly water saturated while the organic-hosted pores may be predominantly gas saturated. These pore-scale changes in wettability result in conditions that may promote the imbibition of both oil-based (hydrophobic) and water-based (hydrophilic) drilling muds, so selecting an OBM or WBM drilling fluid does not necessarily solve the invasion problem.

However, the opposite problem–that of the potential desiccation of cores (water loss)–is greatly reduced in unconventional reservoirs because of the small pore sizes, low porosity and low permeability. Fortunately, this problem can be considerably reduced by appropriate core handling and storage practices.

An analysis of the effects of core storage and processing protocols on measured saturations demonstrates that pore fluids are irreducible at ambient conditions under different storage mechanisms. Even after storing samples in plastic bags for nearly two years, effective porosity, effective water saturation, clay-bound water and permeability measurements were very similar to the original values recorded on arrival at the laboratory. In addition, the analysis showed that the presence or absence of ambient humidity had minimal effect on measured saturations.

Retort Fluid Extraction

The retort fluid extraction method using crushed rock for tight shales offers interesting advantages in relation to conventional methods. Not only does it have the ability to separate free from bound fluids (water and oil) and directly measure the extracted oil and water volumes (API Recommended Practice 40), but it also allows fluid extraction to be completed within a relatively short time. In fact, retort fluid extraction on crushed rock can be completed in a single day, versus up to four weeks for conventional methods

using crushed rock or whole core plugs.

The microstructure of tight shales exhibits a large surface area, significant pore size variability, and a dual-porosity system associated with organic and inorganic constituents. When in contact with liquids, the exposed surfaces support a broad range of interfacial forces (electrical double layer and solvation) that bound the liquids to varying degrees. The pores with organic surfaces (oil wet) and inorganic surfaces (water wet) develop strong capillary forces that bound liquids with strengths that are inversely proportional to the pore radius.

In addition, structurally-bound hydroxyls are also present, and are part of the clay structure. As a result, there is a continuum range of capillary and surface forces keeping water and oil attached to pore surfaces. Extracting these liquids with temperature requires a continuous increase in thermal energy to overcome the bounding forces. During the process, the free or loosely bound water is extracted first at low temperatures, the intermediate water components with various degrees of surface bonding are extracted at continuously increasing temperatures, and strongly bound structural water is extracted last at the highest temperature.

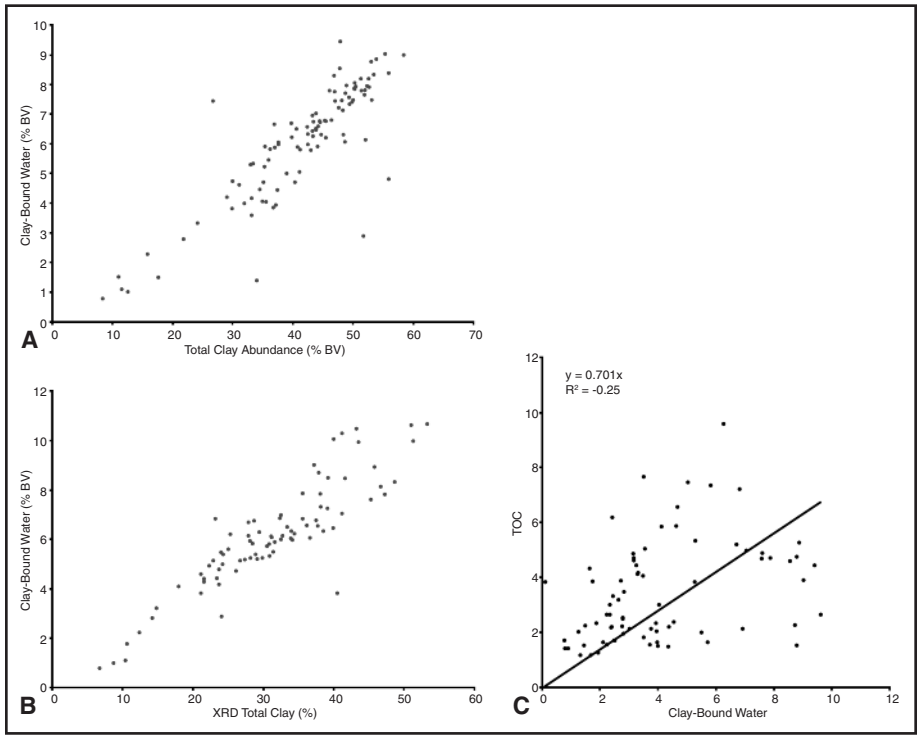
To better characterize the water components produced during retort fluid extraction, retort experiments were conducted on clay standards. These are used for X-ray diffraction (XRD) referencing, and are representative of clays typically present in tight shales. The results illustrate the water extraction behavior associated with these clays and their transformation during dehydration.

Figure 3 shows the amount of water extracted from illite, chlorite, kaolinite and smectite at three temperature steps of 250 (T_1), 600 (T_2) and 1,300 (T_3) degrees Fahrenheit, normalized to the total amount of water ultimately recovered. With the exception of smectite, very little water was extracted from any of the clay standards at the initial retort temperature. In contrast, significant amounts of water were extracted at T_2 and T_3. Given that mature tight shales usually have negligible smectite content, it is inferred that any water produced from an actual reservoir sample at the T_1 temperature was free water.

Figure 3 also shows the XRD mineral abundances of each of the clay species after heating and indicates changes in abundance as a function of progressive

FIGURE 4

Total Clay Abundance (Panel A), XRD Clay Measurements (Panel B) And TOC (Panel C) to Clay-Bound Water



heating. Results show that at the T_1 temperate, the smectite content disappeared, reflecting the conversion of smectite to illite. There was a noticeable decrease in the illite concentration as well. We believe this is because the XRD standard is not pure illite, but mixed illite-smectite clay. Therefore, the decrease in the abundance of the illite-smectite mixture was reflective of the dehydration of the smectite interlayers and conversion to illite. The chlorite abundance remained relatively constant throughout the retort process, while kaolinite abundance remained relatively constant up to the T_3 temperature, where it became amorphous and no longer was detected by XRD.

The early onset and continuous nature of water release in smectite suggests that the retort extraction method cannot separate free water from surface bound water in smectite-rich rocks. The presence of large amounts of smectite is a hallmark of an immature clay system, and is often a proxy for immature kerogen as well. Fortunately, most productive shales and nearly all gas-producing shales are clay mature, or have reasonably low smectite abundances.

Fluid Expulsion Variables

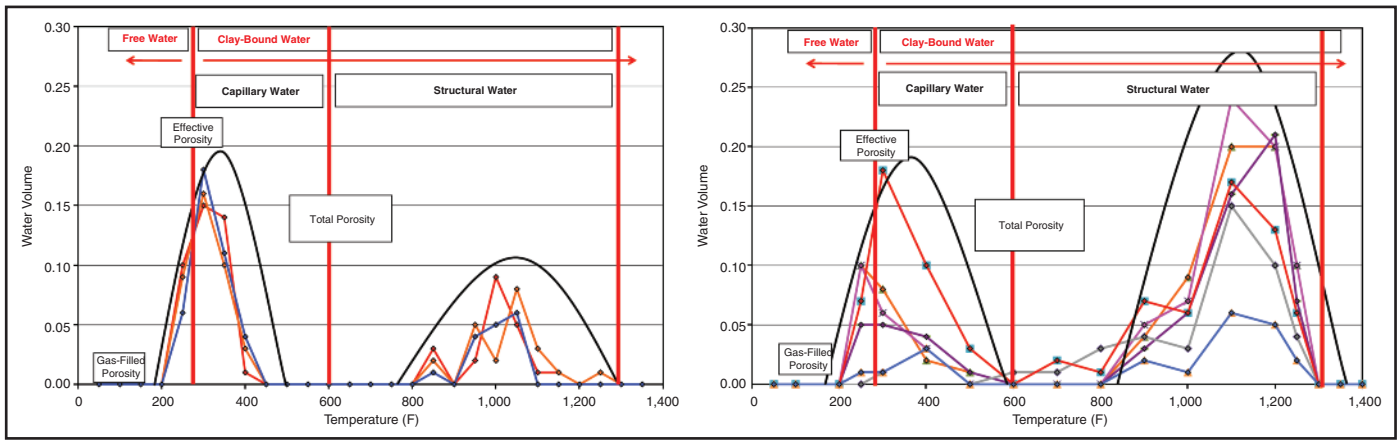
Studies suggest that nonclay minerals do not produce significant quantities of water under retort. This specifically includes quartz and kerogen, the dominant nonclay minerals in many shale gas plays. Panels A and B in Figure 4 show the relationship between clay-bound water abundance (total water at the end of retort minus free water) and XRD measurements of total clay content in two productive shales. The strong correlation in each case suggests that clay is the source of water at higher temperatures.

Panel C in Figure 4 shows the correlation between clay-bound water and kerogen content (TOC). The lack of correlation in this case again suggests that clay is the source of high-temperature retort water. The temperature at which kerogen typically converts to oil is above 400 degrees Celsius, which is considerably higher than either the T_1 (free water) or T_2 (free oil) temperatures. Therefore, any kerogen-derived water obtained at the highest temperature stage would show up as strongly bound or structural water. Yet, we do not see any dependence of clay-bound water on TOC content. This indicates that water generation from organic matter in mature tight shales is not a concern during retort extraction.

Following the experiments on reference clays, and to better understand the gradual

FIGURE 5

High-Resolution Retort Analysis of Siliceous Mudstone (Left) And Argillaceous Mudstone (Right)



evolution of fluid expulsion during the retort process, a set of retort experiments were performed at 50-degree F steps on a series of "typical" mature tight gas shales with little or no smectite abundance.

Figure 5 shows a high-resolution retort analysis of a siliceous mudstone and an argillaceous mudstone. In each plot, the red vertical bars represents the three characteristic retort temperatures (T_1, T_2 and T_3). As temperature increased to T_2, a significant quantity of water was recovered continuously, suggestive of the free water and various capillary, double-layer and surface forces bounding water to solid surfaces. The free water portion is evolved at T_1. Figure 3 shows no water contributed by the clays at this temperature, further suggesting that all the water at T_1 is free.

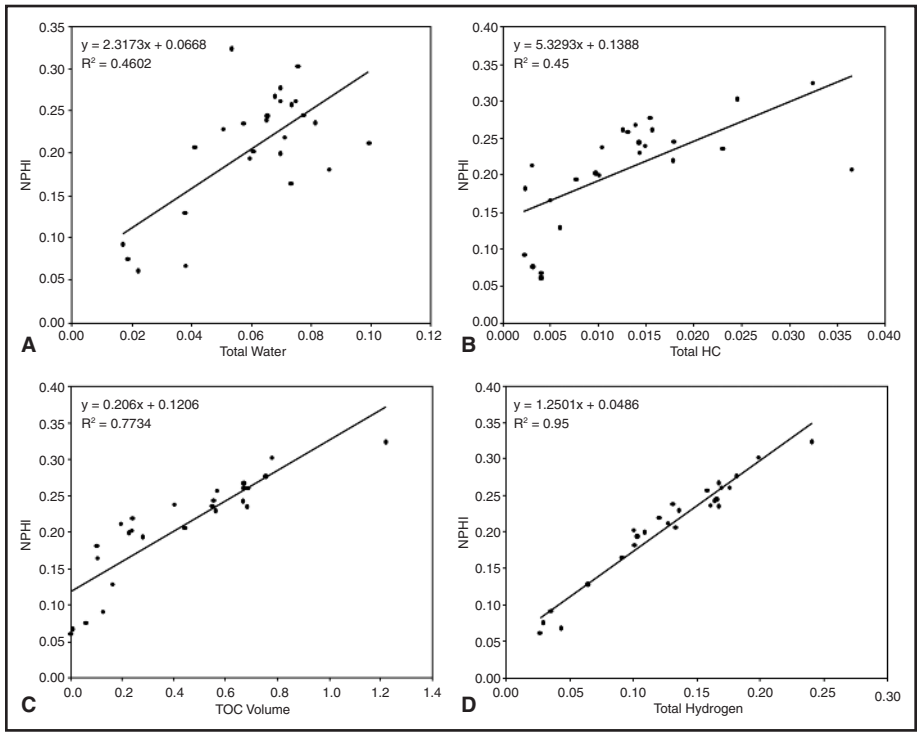
Given that this temperature is slightly above the boiling point of water, any free water should have been extracted at this point. Further temperature increases produced little to no additional water for a range of a few hundred degrees F. However, another slug of water was eventually extracted, and then ceased by T_3. This second slug of extracted water is representative of the strongly bound and structural water.

The retort method also provides a measure of free oil measured at T_2 and surface-bound oil–including bitumen–measured at T_3. Gas saturation is calculated as the portion of the pore space not saturated with either oil or water, free or bound. Therefore, actual measurements of gas saturation are not required. Finally, the volumes of pore-gas, pore-water and pore-oil are used to calculate the pore volume. (For tight shales with TOC content above 4 percent, an additional correction to the pore volume is necessary to account for the volume occupied by the adsorbed gas. This correction is only applicable for the organic-hosted portion of the porosity. It increases with TOC and decreases with increasing pore size).

One interesting advantage of being able to discriminate the various water components during retort extraction is that the measured volumes are used to calculate and discriminate between effective and total porosity, as well as free or free plus capillary-bound/interlayer water saturations. Free water saturation is the free water abundance in effective porosity, while what is commonly referred as "total water saturation" is actually free plus clay-bound water abundance in total porosity. Effective porosity is calculated as gas-filled porosity plus free water extracted at T_1 and oil extracted at T_2. Total porosity is calculated as gas-filled porosity plus free and capillary-bound/interlayer

FIGURE 6

Relationships Among Recovered Water (Panel A), Oil Phase (Panel B), TOC (Panel C) and Neutron Porosity Log (Panel D)



water extracted at T_2, and oil extracted at the same temperature.

Neutron Porosity Data

Neutron porosity data from a wireline log were compared to results from a suite of retort fluid extraction samples from a thermogenically mature, argillaceous, tight gas shale reservoir in the United States. For each core sample with retort analysis, the corresponding neutron porosity log value was evaluated at the corresponding depth (i.e., corrected for the core-to-log shift using corresponding core and log gamma ray curves).

The neutron log does not make any distinction of the hydrogen source, which may reside in the fluid phases (oil and water), minerals or kerogen. Oil and water have about the same hydrogen abundance, while gas has very low density and imparts very little contribution to the hydrogen signal captured by the log. Various minerals–including clays–may contain surface-bound water and structural hydroxyl groups. Kerogen is a strong source of hydrogen, which changes as a function of kerogen maturation.

The neutron porosity value from the log was compared separately with the total amount of water recovered at T_3, and the total organic carbon content of the rock (Figure 6). The volumes reported on the X-axis of each plot were real measured volumes from actual samples. The results show that these components taken individually do not provide a strong relationship between water or hydrocarbon saturation and neutron porosity (hydrogen content). The relationship improves somewhat when comparing TOC and neutron porosity. However, when all three components are summed together, a strong correlation arises between neutron porosity and total hydrogen, as defined by the hydrogen contributions of water, oil and kerogen.

Because of the relatively higher volumetric abundance of kerogen compared with the liquid phases, one might suggest that the strong correlation between total hydrogen in Figure 6 is predominantly driven by the TOC and not by fluid saturations. To address this concern, random values were assigned to the water and oil volumes scaled to their measured magnitudes, and the TOC volume was kept the same. The resulting comparison of total hydrogen with the neutron porosity log values shows that the relationships and trend are considerably weaker than for the measured data, suggesting that the liquid saturations do play a significant role.

Principal Goal

Tight shale formations are difficult to analyze using traditional core analysis methods. Unfortunately, their colloidal size sediments, planar shape grains, the mixture of organic and inorganic components, and weak contacts result in extensive microcracking and the generation of partings. As a result, the principal goal of most measurements on tight shales is to minimize the effect of coring-induced artifacts, with sample crushing being the most consistent method to eliminate microcracking.

Traditional saturation methods have been modified and run on crushed rock for tight shale applications. This has the advantage of considerably reducing the time for fluid extraction while significantly increasing the accuracy of the results. Advanced retort measurements provide highly accurate and reliable measurements of extracted fluids. They have been validated using clay standards and measurements with small temperature increments and semicontinuous fluid extraction.

Research is shedding additional light on the presence and distribution of the various water components (free, clay bound and structural). The new method provides direct measurements of water and oil. It also discriminates free and bound oil and water components, and gives a better understanding of the associated relationships between effective and total porosities.

Total hydrogen content, as measured by the neutron log, is strongly correlated to the total volume of water, oil and kerogen. This includes free, bound, and structural components. The results demonstrate the accuracy and value of the neutron log measurements for detecting total hy-

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drogen, and also show that the strong relationship from these two independent measurements can be used to validate the extracted volumes.

Although this discussion is centered on evaluating mature tight shale systems that are predominantly in the dry gas window, evaluating oil-producing mudstones presents additional complications related to the increased liquid hydrocarbon content and the phase transformations associated with pressure and temperature changes. Laboratory saturation conditions for oil-producing shales, even under no invasion during drilling, are substantially different than native saturation conditions, although we have observed that water saturations are generally consistent, regardless of the hydrocarbon generated. This implies that an accurate hydrocarbon saturation and hydrocarbon porosity can still be obtained from retort.

The additional complication comes in determining oil composition and oil saturation in situ as a function of phase changes caused by lowering pressure and temperature while transporting the core to the surface. Sampling and obtaining a complete characterization of the hydrocarbon species in situ for pressure/volume/ temperature and phase transformation analysis is challenging. A substantial research effort focused on these issues is under way. ❐