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ABSTRACT

Experimental studies have been accomplished aimed at studying the formation factor of a partially saturated rock. The effective formation factors to an electrolyte solution of the pore spaces not occupied by a wetting fluid (paraffin wax) have been measured at various saturations, after solidifying the wetting fluid in place. The experimental data is studied in light of the role of the pore structure on the wetting fluid invasion process with the aid of fluid distributions at each saturation regime, a complete rock pore cast, and its associated rock section. The effect of clay minerals on formation factor is studied. The surface conductance contribution of clay minerals to overall electrical conductivity is assessed. The effect of partial hydrocarbon saturation on overall rock conductivity and on the Archie saturation exponent is discussed.

I. INTRODUCTION

The transport properties of porous rocks such as hydraulic and electrical conductivity are determined by pore topology, geometry, and the physics and chemistry of mineral-fluid and fluid-fluid interactions. A primary focus of our research is to understand with analysis and experiment how these factors affect the property under consideration. On our electrical conductivity experimental studies, we have used a wetting fluid that can be frozen in place: 1) to allow measurement of effective properties to an electrolyte solution of the rock pore spaces not occupied by a wetting fluid, 2) to allow direct observation and analysis of the wetting fluid distributions at each fluid saturation regime, and 3) to understand how the wetting fluid invasion process is controlled by rock pore structure and topology with the aid of a complete pore cast. In addition, disseminated clay, often in the form of aggregates, may line the rock pores or be distributed between the grains of the matrix. The clay minerals act as a separate conducting path additional to that contributed by the saline solution in the rock pores. The conductivity of the rock containing disseminated clay therefore has two components: one is the conductivity of the fluid-filled pores of the rock; the other one is the conductivity of the disseminated clays which are the solid phases that primarily exhibit surface reactivity in rocks and soils (Sposito, 1984). This is a direct consequence of the fact that clay particles have large surface areas. In order to measure surface conductance, the geometry of the system must be known. In the case of rocks, the exact pore geometry that affects the total conductance is unknown. Furthermore, it can change due to dispersion and flocculation of the clay minerals in the pores. Thus, it is foreseen that the presence of clay may greatly complicate the quantitative evaluation and interpretation of experimental data. In our experimental studies, we have isolated the effect of clay on formation factor and assessed the surface conductance contribution due to the presence of clays.

The conductivity of a rock (consisting of electrically nonconductive particles) saturated with a conducting electrolyte has been shown by Archie (1942) to depend upon the conductivity of the electrolyte and the geometrical (e.g., formation) factor. In such a system, the electrical conductance of the saturated rock is proportional to the conductance of the electrolyte solution; the constant of proportionality (always > 1) is a geometrical factor called the formation factor. The formation factor of a fully saturated rock is defined as

$$F = \frac{\rho(\text{electrolyte saturated sample})}{\rho(\text{electrolyte solution})} \quad (1)$$

where F is the formation factor, and ρ the resistivity.

The assumptions implicit in the formation factor concept are: 1) The conduction process is electrolytic and it occurs only through the network of saline solution filling

the pores, 2) the properties of the saline solution filling the pores are uniform, and 3) there are no surface conductivity effects. In such a system, the plot of the electrical conductance of the saturating solution versus the electrical conductance of the rock saturated with the solution is a straight line passing through the origin.

When more than one fluid is present, e.g., oil/water, the empirically determined Archie saturation is often found to vary according to (Archie, 1942; King et al., 1988)

$$F = \phi^{-m} S_w^{-n} \quad (2)$$

where F is the formation factor, ϕ is the porosity, S_w the electrolyte saturation, and m and n dimensionless empirically-determined constants. The value of m varies with the extent to which a rock is cemented, from about 1.3 to 2.3 (Wyllie, 1963). The value of n depends to a large extent on the wettability of a reservoir rock (Anderson, 1986). In rocks that are wholly oil-wet, i.e., rocks in which the water is the non-wetting phase, n ranges from 3 to about 4. If the rock surface is entirely water-wet, the value of n lies between 1.8 and 2.0 (Wyllie, 1963). The main assumptions implicit in the Archie saturation equation are: 1) the saturation/resistivity relation is unique (i.e., only one resistivity is measured at a given saturation), 2) n is constant for a given porous medium, and 3) all the saline solution contributes to the flow of electric current. To test these assumptions, we have analyzed the electrical conductivity experimental data in light of the role of the pore structure and topology in the wetting fluid invasion process with the aid of the fluid distributions at each saturation regime and a complete pore cast.

Laboratory electrical conductivity data for fully and partially-saturated samples of Berea sandstone with a wetting fluid is presented. In our studies, we use a wetting fluid (paraffin wax) that can be frozen in place at controlled saturations to allow us to examine the occupied pore space after the experiment. The effective formation factors for an electrolyte in the pore spaces not occupied by the paraffin are measured at various saturations after solidifying the paraffin in place. The effect of clay and other surface reactive minerals on Berea sandstone formation factor is first isolated and their surface conductance contribution to overall conductivity assessed. The electrical conductivity experimental data is analyzed in light of the role of the pore structure on the wetting fluid invasion process with the aid of direct observation of fluid distributions at each saturation regime, a complete pore cast, and its corresponding rock section. Finally, the effect of partial hydrocarbon saturation on overall Berea sandstone conductivity, and on the Archie saturation exponent n is studied.

II. APPARATUS AND PROCEDURE

The apparatus that was used in this study was designed to simultaneously measure both hydraulic and electrical conductivity (Fig. 1) (Schlueter et al., 1992). The rock core of 5 cm in diameter and 5 cm in length was encased in its rubber jacket and placed in the test cell. The test cell base was connected directly to the bottom of the sample and had a centrally located orifice to allow fluids to flow through the mounted core. The electrodes were connected to the top and bottom of the sample so that any current flow between them necessarily passed through the core. A confining pressure of 50 psi (3.4 atm) was applied via nitrogen gas. Fluid flow through the core was controlled by a syringe pump providing a constant flow rate of 200 ml/hr. The basic procedure used for measuring the formation factor in a 100% electrolyte saturated sample was to first fully vacuum saturate each core with distilled water. An aqueous zinc nitrate solution of known resistivity was then flowed through the core and continued for sufficient time to establish constant pressure and resistance readings. It was found that about four pore volumes of electrolyte were needed to achieve steady state. In our experiments, we used samples of Berea sandstone, which is a homogeneous sedimentary rock used as a reference rock in the petroleum industry. The intrinsic permeability and porosity of a Berea sandstone core are about 600 md ($600 \times 10^{-15} \text{ m}^2$) and 22%, respectively. It is estimated to be of Mississippian age and is found in Berea, Ohio.

III. EFFECT OF PARTIAL HYDROCARBON SATURATION ON FORMATION FACTOR

The conductivity of the pore system can be varied either by altering the salinity of the water in the pores or by changing the quantity of water of a particular salinity by introducing another solid/fluid phase into the pore space. To determine the effect of partial fluid saturation on formation factor, we utilized Berea sandstone samples that had been permeated with a sequence of triple distilled water (to measure the hydraulic conductivity) followed by a 0.5 M zinc nitrate solution and again flushed with distilled water. This procedure was applied to every sample to find the formation factor before the paraffin application (Table I). The samples were then oven dried. After measuring the formation factor, the samples were partially filled with paraffin wax at controlled saturations of 20%, 40%, 50%, 60%, and 69%. The hydrocarbon paraffin is a wetting phase with a density of 0.76 g/cm^3 , and a melting point of 56°C . It is applied in the rock at temperatures higher than its melting point until uniform saturation is achieved throughout the sample. The paraffin is then solidified in place at ambient temperature conditions. After the hydrocarbon paraffin application, the rock grain surfaces became hydrophobic. To measure the effective formation factor, the rock

samples that were partially saturated with paraffin were permeated with a 0.5 M zinc nitrate solution at pH $\sim$ 4. The effective formation factor vs. paraffin saturation is presented in Fig. 2 (Case A). Experiments in which the formation factor had not been measured previous to paraffin impregnation were repeated on a new and clean set of samples. The result is shown in Fig. 2 (Case B). In summary, good agreement within experimental error was found between the two sets of experiments A and B.

IV. EFFECT OF CLAY ON FORMATION FACTOR

Berea sandstone rock contains about 10% clay minerals coating the available pore space. X-ray diffraction studies by Khilar and Fogler (1984), in conjunction with scanning electron microscopy (Fig. 3) and energy dispersive x-ray analysis (Fig. 4), indicate that Berea sandstone contains about 8% by weight of dispersible and swelling clays (mainly kaolinite with some illite, and smectite), 80% quartz, and 12% feldspar. To reduce the tendency for the clays to disperse, a bivalent cation such as zinc was used. For solutions with cations of valence > 1 , at pH 7, no critical salt concentration below which clay is released from Berea sandstone pore walls has been found (Khilar and Fogler, 1984). It is important to recognize that the clay minerals present in the rock have been immobilized by the application of the hydrocarbon paraffin coating the pores. Therefore, the formation factor extrapolated to electrolyte saturation of unity (i.e., no hydrocarbon paraffin) corresponds to the formation factor of the clean rock without clays and without any surface conduction effects caused by the surface reactive minerals that may be present in the rock. The formation factor at paraffin saturation of zero, $F=18$ (Fig. 2), has been extrapolated from a plot of the multiplicative inverse of the residual formation factor vs. paraffin saturation. This hypothesis was verified by partially removing clays in a Berea sandstone core with an acid treatment of a mixture of 6% hydrochloric and 1.5% hydrofluoric acids (Suarez, 1991). After the core was treated and clays flushed out, the formation factor was found to be 16.4, larger than the average formation factor of 15.7 that was measured for samples containing clay (Table I).

V. EFFECT OF SURFACE CONDUCTANCE ON FORMATION FACTOR

To investigate the magnitude of surface conductance contribution due to clays and other surface reactive minerals on formation factor, we investigated the influence of solution concentration on rock electrical conductivity. For that purpose, resistivity experiments were performed on two Berea sandstone cores. The first core was permeated with solutions of zinc nitrate at increasing concentrations of 0.05 M, 0.1 M, and 0.5 M while measurements of resistivity were taken. After equilibrium had been

reached with one solution, another zinc nitrate solution of less resistivity was flowed through the core, and a constant resistance reading was again obtained. To obtain a more complete set of electrical conductivities measurements at low electrolyte concentrations, the procedure was repeated on a new core with solutions that included salinities 0.005 M and 0.01 M zinc nitrate (pH of the effluent was found to vary from ~ 4.5 to ~ 4.0 , respectively). The data thus obtained enabled the formation factor of the rock to be computed, and also confirmed the ability of an invading fluid to displace interstitial water from a rock core. Results are given in Table II. The conductivity of the fluid (κ_w) vs. the conductivity of the fully saturated rock (κ_r) data are plotted in Fig. 5. As observed, minor deviations from the Archie formation factor concept are present at low electrolyte concentration. There is a rapid initial increase in rock conductivity as the solution concentration increased up to about 0.005 M. When the solution in equilibrium was more concentrated than 0.005 M, there was a decrease in rock conductivity with an increase in solution conductivity until a solution concentration of about 0.05 M. Thereafter, the formation factor is constant at least over the range of concentrations studied. The shape of the ($\kappa_w - \kappa_r$) curve at low electrolyte concentrations may be interpreted in light of the mechanisms and factors controlling clay-fluid interactions as they relate to clay-water electrolytic conduction. Berea sandstone contains mainly kaolinite clay. At $4.0 < \text{pH} < 4.5$, and electrolyte concentrations lower than 0.01 M, experiments in kaolinite clay by Williams and Williams (1978) (using sodium chloride as electrolyte) (Fig. 6), show that an increase in solution concentration produces a decrease in ion mobility, and thus a reduction in electrical conductivity. In addition, for clay-water systems, an increase in salt concentration is expected to compress the diffuse double layer, to decrease the electrical interaction between the cations and the clay surface, and to reduce the electrical conductivity. At higher electrolyte concentrations the double layer compresses further, there is a more energetic conductive path through the solution, and the ($\kappa_w - \kappa_r$) plot becomes linear.

The trend of change in rock electrical conductivity at low electrolyte concentrations does reflect the contribution to surface conduction of clays and other surface reactive minerals. However, our experimental results show that for Berea sandstone, the surface conduction component represents a minor contribution to overall electrical conductivity, and therefore can be ignored for most applications.

VI. EFFECT OF PORE STRUCTURE AND TOPOLOGY ON EFFECTIVE FORMATION FACTOR

To understand how pore structure and topology control the transport property of consideration, the electrical conductivity data (Fig. 2) have been studied in light

of the wetting fluid distributions at each saturation regime (Figs. 7, 8, and 9) with the aid of a complete rock pore cast (Fig. 10) and its associated rock section (Fig. 11). The rock pore cast was obtained from a rock specimen which had been fully impregnated with Wood's metal alloy and the quartz grains removed by hydrofluoric acid. The rock pore cast and its associated rock section clearly reveal that the pore space is composed of grain contact porosity (e.g., thin sheets and micropores), and intergranular porosity. Figure 7 shows a scanning electron microscope micrograph collage of a Berea sandstone specimen that has been partially saturated with about 20% – 30% paraffin. The gray phase corresponds to quartz grains, the white phase corresponds to pores that have been impregnated with paraffin, and the black phase corresponds to the remaining pore space filled with blue epoxy for imaging purposes. The paraffin has invaded grain contact pore space (e.g., thin sheets and micropores) and intergranular pore space connected by smaller throats while only coating available intergranular channels connected by larger throats. A substantial effect on effective formation factor is observed. Thus, the fraction of the pore structure connected by smaller constrictions provides important alternative routes to intergranular conduits connected by larger throats for the ions to travel. Figure 8, displays a scanning electron microscope collage of a rock specimen partially saturated with about 40% – 50% paraffin. At this stage, we are filling intergranular conduits connected by the larger throats. Also, a portion of the electrolyte has apparently lost continuity as the paraffin saturation is increased over $\sim 30\%$ so that the resistivity increased at a faster rate. Hence, a larger effect on effective formation factor is observed. Figure 9, displays a scanning electron microscope collage of a rock specimen partially saturated with about 60% - 70% paraffin. We have filled almost all intergranular conduits connected by larger throats. A few intergranular pores not well connected still remain unfilled. When paraffin saturation is $\sim 70\%$ the whole pore structure behaves as disconnected.

VII. THE ARCHIE SATURATION EXPONENT

Electrical conductivity data presented in Fig. 2 (Case B) have been replotted on a logarithmic scale for the effective formation factor vs. electrolyte saturation (Fig. 12). To understand their physical significance, the plot has been divided in three zones: zone I, a linear zone between saturations of the electrolyte of about 0.7 (S_{crit}) and 1, with an Archie exponent of $n \sim 3$; zone II, a linear zone between electrolyte saturations of about 0.5 and 0.7, with an Archie exponent of $n \sim 5$; and zone III, a zone for electrolyte saturations less than 0.5. Zone I, with Archie saturation exponent of ~ 3 , reflects the fact that grain contact pore space (e.g., thin sheets and micropores), and intergranular pore space connected by the smaller throats provide important alternate paths to intergranular conduits connected by larger throats for

the ions to travel. Zone II, with Archie saturation exponent of ~ 5 , reflects the fact that as the electrolyte saturation is lowered below S_{crit} part of the rock structure composed of pores connected by the smaller throats becomes inactive. In addition, intergranular conduits connected by larger throats start being filled and partially filled with paraffin. Thus, part of the electrolyte available for the transport of ions loses continuity and the resistivity is increased at a faster rate.

VIII. SUMMARY

The formation factor of a partially saturated rock with a wetting phase is controlled by the rock structure and topology as well as the physics and chemistry of mineral-fluid interactions. To understand the relationships, we have measured the effective formation factor to an electrolyte of the pore spaces not occupied by a wetting fluid (paraffin), after solidifying the fluid in place. It is important to recognize that when the rock is partially saturated with the hydrocarbon paraffin, the clay minerals present in the rock pore space are immobilized. Thus, the formation factor extrapolated to electrolyte saturation of unity (and paraffin saturation of 0), $F = 18$, corresponds to the formation factor of the 'clean' rock (without clay). Eventhough the change in trend of rock electrical conductivity at low electrolyte concentrations does represent the contribution of surface conduction due to clays, this contribution is negligible. The effective formation factor data have been studied in light of the wetting fluid distributions observed at different saturations levels with the aid of a complete pore cast and its associated rock section. Our analysis shows that: 1) $\sim 30\%$ of the pore space comprises grain contact pores (e.g., thin sheets and micropores), and intergranular pores connected by smaller throats, 2) $\sim 40\%$ of the pore space comprises intergranular conduits composed of pores connected by larger throats, and 3) $\sim 30\%$ of the intergranular pore space remains disconnected. The grain contact pore space of large surface area (thin sheets), micropores and intergranular pores connected by smaller throats provide important alternate paths to the intergranular conduits connected by larger throats for the ions to travel. Therefore, for a consolidated rock such as Berea sandstone, we find no unique relationship between effective formation factor and electrolyte saturation, and no unique definition of the Archie saturation exponent, n , for the full range of saturation. Finally, Archie saturation exponent n is found to vary from approximately 3 when connected grain contact pore space (e.g., thin sheets and micropores), and intergranular pores connected by smaller throats are filled with hydrocarbon paraffin to approximately 5 when intergranular conduits connected by the larger throats are filled with hydrocarbon paraffin, with a critical saturation (S_{crit}) of 0.7.

IX. ACKNOWLEDGMENTS

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TABLE I. Formation Factor Data - Berea Sandstone, with a 0.5 M zinc nitrate solution used as electrolyte.

Sample	ϕ^a (%)	F ^b
KS4	21.9	15.0
KS6	22.6	14.3
KS7	22.0	17.7
KS9	22.2	15.9

^a porosity.

^b formation factor.

TABLE II. Resistivity Data - Berea Sandstone, with zinc nitrate solution used as electrolyte.

M ^a	pH (effluent)	ρ_w^b (ohm-m)	ρ_r^c (ohm-m)
0.5	—	0.19	3.35
0.1	—	0.68	10.4
0.05	—	1.24	20.1
0.01	4.0	5.08	73.9
0.005	4.5	9.01	101.3

^a solution molarity.

^b solution resistivity.

^c rock resistivity.

FIG. 1. Schematic representation of the laboratory apparatus designed to simultaneously measure both electric and hydraulic conductivity.

FIG. 2. Effective formation factor vs. hydrocarbon paraffin saturation for Berea sandstone. The pore space was partially saturated with hydrocarbon paraffin. The remaining portion of the pore space was filled with a 0.5 M zinc nitrate solution. Cases A and B correspond to experimental data on samples subjected, or not subjected, to a formation factor measurement previous to paraffin application, respectively.

FIG. 3. Scanning electron micrographs of a Berea sandstone specimen showing the presence of fine-grained clay minerals (mainly kaolinite) partly filling pores between quartz grains.

FIG. 4. X-ray spectrum of clay minerals coating Berea sandstone sample pores shown in figure 3. EDX analysis yielding nearly equal peak heights of Si and Al confirms the identification as kaolinite.

FIG. 5. Conductivity data of Berea sandstone at different electrolyte concentrations. Zinc nitrate electrolyte solutions with concentrations ranging from 0.005 M to 0.5 M were used.

FIG. 6. Mobility for kaolinite as a function of salt concentration and pH (after Williams and Williams, 1978).

FIG. 7. Scanning electron micrograph collage of a Berea sandstone specimen impregnated with about 20% - 30% paraffin. The actual width of field is approximately 6 mm. The gray phase is quartz grains, the white phase is pores saturated with paraffin, and the black phase is remaining pores filled with blue epoxy for imaging purposes.

FIG. 8. Scanning electron micrograph collage of a Berea sandstone specimen impregnated with about 40% - 50% paraffin. The actual width of field is approximately 6 mm. The gray phase is quartz grains, the white phase is pores saturated with paraffin, and the black phase is remaining pores filled with blue epoxy for imaging purposes.

FIG. 9. Scanning electron micrograph collage of a Berea sandstone specimen impregnated with about 60% - 70% paraffin. The actual width of field is approximately 6 mm. The gray phase is quartz grains, the white phase is pores saturated with paraffin, and the black phase is remaining pores filled with blue epoxy for imaging purposes.

FIG. 10. Scanning electron micrograph collage of a Berea sandstone pore cast. The actual width of field is approximately 6 mm. The rock pore space was filled with Wood's metal alloy, and the quartz grains removed by hydrofluoric acid to allow direct observation of the pore structure.

FIG. 11. Scanning electron micrograph collage of a Berea sandstone rock specimen fully impregnated with Wood's metal alloy. The actual width of field is approximately 3 mm. The gray phase is quartz grains, and the white phase is pores saturated with the alloy. The section reveals that the pore space is composed of grain contact (e.g., thin sheets and micropores), and intergranular porosity.

FIG. 12. Effective formation factor vs. electrolyte saturation for Berea sandstone (Case B). The pore space was partially saturated with hydrocarbon paraffin, with the remainder of the pore space filled with a 0.5 M zinc nitrate solution.

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