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Formation Factor and the Microscopic Distribution of Wetting Phase in Pore Space of Berea Sandstone

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Transport properties of porous rocks, such as hydraulic and electrical conductivity, are determined by pore geometry, topology, and the physics and chemistry of mineral-fluid and fluid-fluid interactions. A primary focus of our research is to understand, through analysis and experiment, how these factors affect the macroscopic property under consideration. In our experimental electrical conductivity studies, we have employed a wetting fluid that can be frozen in place: (1) to allow measurement of effective properties with an electrolyte solution in the rock pore spaces not occupied by a wetting fluid, (2) to allow direct observation and analysis of the wetting-fluid distribution 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 rock pores or be distributed between matrix grains. Clay minerals are the solid phases that primarily exhibit surface reactivity in rocks and soils (Sposito, 1984). In our investigation, we have isolated the effect of clay minerals on formation factor and assessed their surface conductance contribution. The formation factor of a fully saturated rock is the ratio of the resistivity of an electrolyte-saturated core sample to the resistivity of the electrolyte solution. When more than one fluid is present in the rock pore space, e.g., oil/water, the empirically determined Archie saturation is often found to vary according to (Archie, 1942)

$$F = \phi^{-m} S_w^{-n},$$

where F is the effective formation factor, ϕ is porosity, m and n are dimensionless empirically determined constants, and S_w is the electrolyte saturation. The value of m varies with the degree of rock cementation, from about 1.3 to 2.3 (Wyllie, 1963). The value of n depends to a large degree on the wettability of a reservoir rock (Anderson, 1986). In rocks that are wholly oil-wet, i.e., rocks in which water is the nonwetting phase, $3.0 < n < 4.0$, whereas if the rock surface is entirely water-wet, $1.8 < n < 2.0$ (Wyllie, 1963). The Archie saturation equation assumes that the saturation/resistivity relation is unique, n is constant for a given porous medium, and all the saline solution contributes to the flow of electric current. To verify these assumptions, we have analyzed the experimental electrical conductivity data in light of the role of pore structure in the wetting-fluid invasion process with the aid of fluid distributions at each saturation regime, a complete pore cast, and its associated rock section.

APPARATUS AND PROCEDURE

The apparatus used in this study was designed to measure simultaneously both hydraulic and electrical conductivity (Schlueter et al., 1992). The rock core (5 cm in diameter and 5 cm in length) is encased in its rubber jacket and placed in the test cell. The test cell base is connected directly to the bottom of the sample, and a centrally located orifice is attached to allow fluids to flow through the mounted core. The electrodes are connected to the top and bottom of the core sample so that any current flow between them will necessarily pass through the core. A confining pressure of 50 psi (3.4 atm) is applied using nitrogen gas. Fluid flow through the core is 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 is first to vacuum saturate each core completely with distilled water. An aqueous zinc nitrate solution of known resistivity is then pumped through the core, and flow is continued for a sufficient time to establish constant pressure and resistance readings. It was found that about four pore volumes of electrolyte is required to achieve steady state. In our experiments, we used samples of Berea sandstone, a homogeneous sedimentary rock of Mississippian age, found in Berea, Ohio, often used as a reference rock in the petroleum industry. The intrinsic permeability and porosity of a Berea sandstone core are about 600 md (600×10^{-15} m^2) and 22%, respectively.

EFFECT OF HYDROCARBON SATURATION

Pore system conductivity can be varied either by altering pore water salinity 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 each sample to discern the formation factor before paraffin application. Samples were then oven dried. After measuring formation factor, samples were partially filled with paraffin wax at predetermined saturations of 20%,

40%, 50%, 60%, and 69%. 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, rock grain surfaces became hydrophobic. To measure effective formation factor, rock samples partially saturated with paraffin were permeated with a 0.5-M zinc nitrate solution at pH ~ 4. The effective formation factor vs. paraffin saturation is presented in Figure 1 (case A). Experiments in which formation factor had not been measured prior to paraffin impregnation were carried out on virgin samples. The result is shown in Figure 1 (case B). In summary, good agreement within experimental error was found between empirical sets A and B.

EFFECT OF CLAY

X-ray diffraction studies by Khilar and Fogler (1984), in conjunction with scanning electron microscopy and energy-dispersive x-ray analysis, indicate that Berea sandstone contains ~ 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 c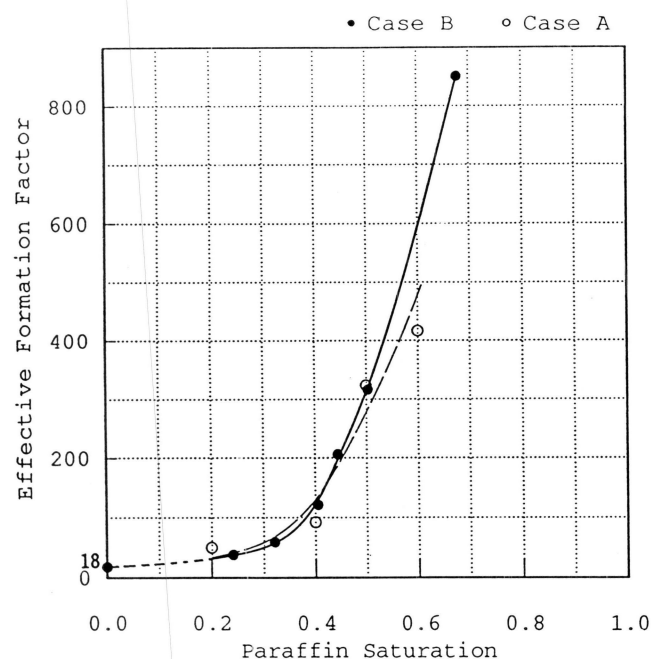lays to disperse, zinc, a bivalent cation, was used. For solutions with cations of valence > 1, at pH = 7, no critical salt concentration has been found below which clay is released from Berea sandstone pore walls (Khilar and Fogler, 1984). It is important to recognize that clay minerals present in the rock have been immobilized by coating pores with hydrocarbon paraffin. Therefore, formation factor extrapolated to an electrolyte saturation of unity (i.e., no hydrocarbon paraffin) corresponds to that of clean rock without clays and without surface conduction effects caused by the pressure of surface reactive minerals. Formation factor at a paraffin saturation of zero, $F = 18$ (Figure 1), 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 by acid treatment with a mixture of 6% hydrochloric and 1.5% hydrofluoric acids (Suarez-Rivera, personal communication, 1991). After the core was treated and clays flushed out, formation factor was found to be 16.4, larger than the average formation factor of 15.7, measured for samples containing clay.

EFFECT OF SURFACE CONDUCTANCE

To study the magnitude of surface conductance contribution due to clays on formation factor, we investigated the influence of solution concentration on rock electrical conductivity. Resistivity experiments were performed on Berea sandstone cores. Rock specimens were permeated with solutions of zinc nitrate at increasing concentrations from 0.005 M to 0.5 M while resistivity measurements were taken (Table 1). After equilibrium had been reached with one solution, another zinc nitrate solution of lower resistivity was flowed through the core, and a constant resistance reading was again obtained. The data thus obtained permitted 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. The trend of change in rock electrical conductivity at low electrolyte

Figure 1. Effective formation factor vs. 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 before paraffin application, respectively. [XBL 934-429]

Table 1. Resistivity data for Berea Sandstone with zinc nitrate solution saturating the sample.

M^a	pH (effluent)	ρ_w (ohm-m)[b]	ρ_r (ohm-m)[c]
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.

concentrations reflects the contribution to surface conduction of clays. However, experimental results show that for Berea sandstone, the surface conduction component due to clays is a minor contribution to overall electrical conductivity and therefore can be ignored for most applications.

EFFECT OF PORE STRUCTURE

To understand how pore structure and topology control the transport property under consideration, electrical conductivity data (Figure 1) have been studied in light of the wetting-fluid distributions at each saturation regime (Figures 2 to 4) with the aid of a complete rock pore cast and its associated rock section (Figure 5). The rock pore cast was obtained from a rock specimen that 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 (thin sheets and micropores) and intergranular porosity. Figure 2 shows a scanning electron microscope (SEM) photomicrograph collage of a section of Berea sandstone that has been partially saturated with approximately 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, which was filled with blue epoxy for imaging purposes. Paraffin has invaded grain-contact pore space (i.e.,
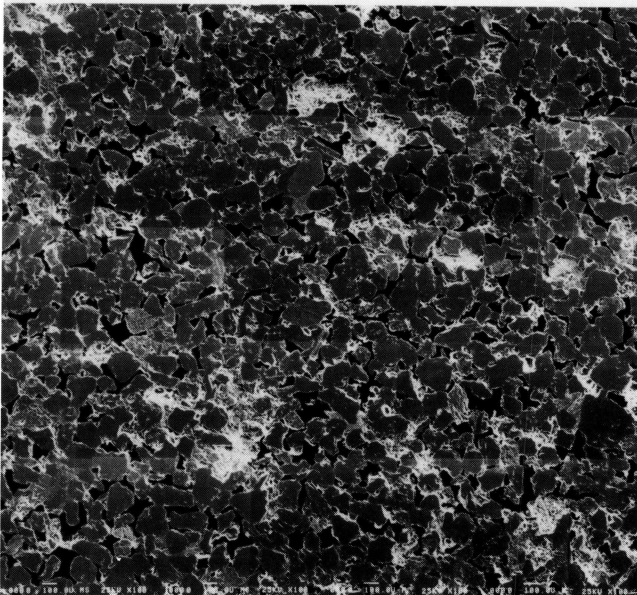

Figure 3. SEM photomicrograph collage of a Berea sandstone specimen impregnated with approximately 40–50% paraffin. The actual width of field is about 4.5 mm. The gray phase is quartz grains, the white phase is pores saturated with paraffin, and the black phase is remaining pores filled with blue epoxy for imaging purposes. [XBB 925-3164]

Figure 2. SEM photomicrograph collage of a Berea sandstone specimen impregnated with approximately 20–30% paraffin. The actual width of field is about 4.5 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. [XBB 926-4990]
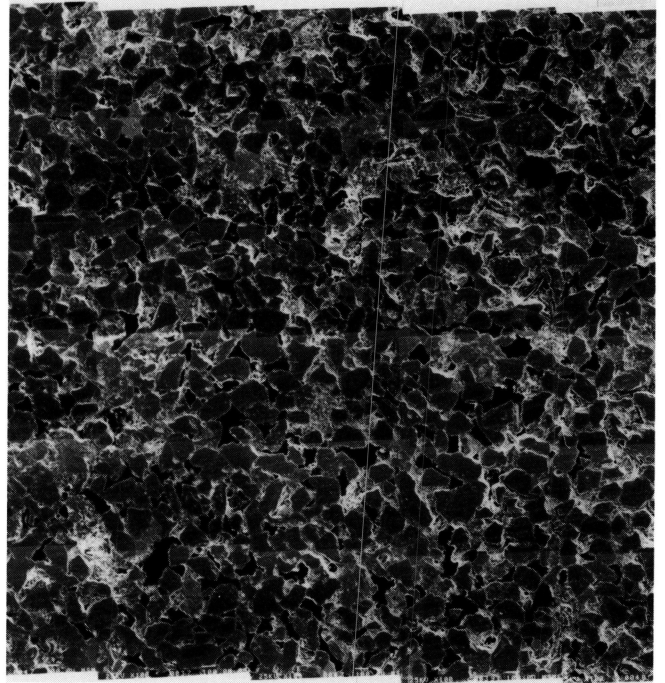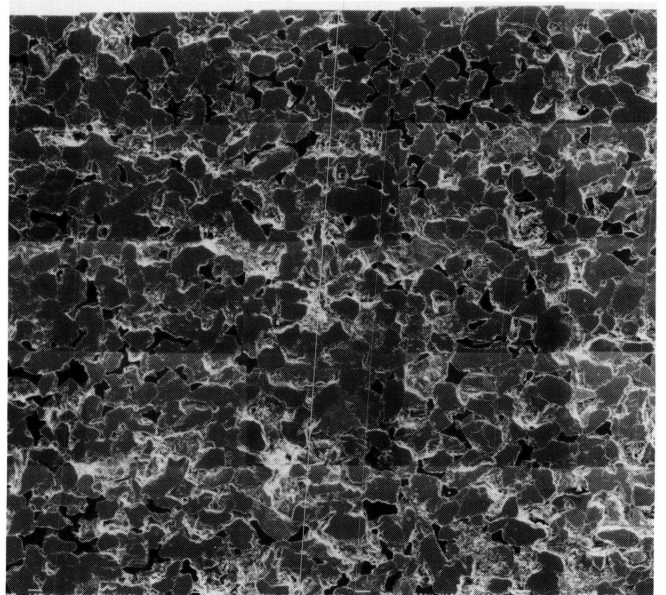

Figure 4. SEM photomicrograph collage of a Berea sandstone specimen impregnated with approximately 60–70% paraffin. The actual width of field is about 4.5 mm. The gray phase is quartz grains, the white phase is pores saturated with paraffin, and the black phase is remaining pores filled with blue epoxy for imaging purposes. [XBB 924-2700]
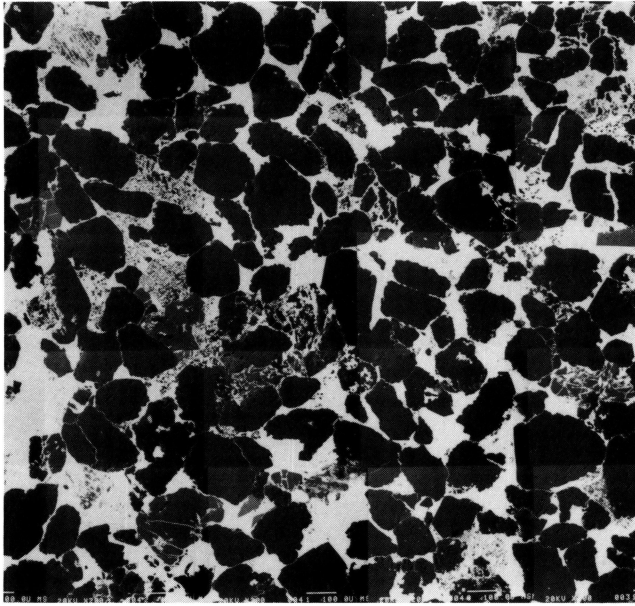
Figure 5. SEM photomicrograph collage of a Berea sandstone rock specimen fully impregnated with Wood's metal alloy. The actual width of field is about 2.3 mm. The gray phase is quartz grains, the white phase is pores saturated with the alloy. The section reveals that the pore space is composed of grain-contact porosity (i.e., thin sheets and micropores) and intergranular porosity. [XBB 932-1806]

Figure 6. 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. [XBL 934-430]

thin sheets and micropores) and intergranular pore space connected by smaller throats but has only coated the available intergranular channels connected by larger throats. A strong effect on effective formation factor is observed. Therefore, the fraction of the pore structure connected by smaller constrictions provides the ions with important alternative routes to intergranular conduits connected by larger throats. Figure 3 shows an SEM photomicrograph collage of a rock section partially saturated with approximately 40–50% paraffin. At this stage, we are filling intergranular conduits connected by the larger throats, and a portion of the electrolyte has apparently lost continuity as the paraffin saturation is increased over about 30%, so that the resistivity increases at a faster rate. A still larger effect on effective formation factor is observed. Figure 4 shows an SEM photomicrograph collage of a rock section partially saturated with approximately 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 ~ 70%, the whole pore structure behaves as though disconnected.

THE ARCHIE SATURATION EXPONENT

Electrical conductivity data presented in Figure 1 (case B) have been replotted on a logarithmic scale for the effective formation factor vs. electrolyte saturation (Figure 6). To un-
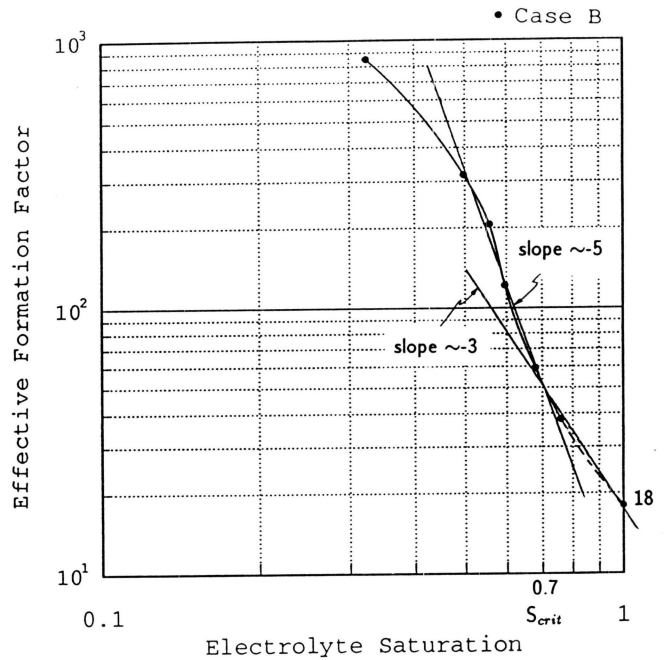
derstand their physical significance, we have divided the plot into three zones: zone I, a linear zone of electrolyte saturations between about 0.7 (S_{crit}) and 1, with an Archie exponent of $n \sim 3$; zone II, a linear zone of electrolyte saturations between about 0.5 and 0.7, with an Archie exponent of $n \sim 5$; and zone III, a zone of electrolyte saturations less than 0.5. Zone I, with an Archie saturation exponent of approximately 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 the ions with important alternate paths to intergranular conduits connected by larger throats. Zone II, with an Archie saturation exponent of approximately 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.

RECAPITULATION

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 with an electrolyte

in the pore spaces not occupied by a wetting fluid (paraffin wax) after solidifying the fluid in place. It is important to recognize that when the rock is partially saturated with hydrocarbon paraffin, clay minerals present in rock pore space are immobilized. Thus formation factor extrapolated to an electrolyte saturation of unity (and paraffin saturation of zero), $F = 18$, corresponds to that of "clean" rock (without clay). Even though the change in the trend of rock electrical conductivity at low electrolyte concentrations represents the contribution to surface conduction due to clays, this component is negligible. Effective formation factor data have been studied in light of the wetting-phase distribution observed at different saturations with the aid of a complete pore cast and its associated rock section. Our analysis shows that (1) ~ 30% of the pore space consists of grain-contact pores (i.e., thin sheets and micropores) and intergranular pores connected by smaller throats, (2) ~ 40% of the pore space consists of intergranular conduits composed of pores connected by larger throats, and (3) ~ 30% of the intergranular pore space remains disconnected. The grain-contact pore space of large surface areas (thin sheets), micropores, and intergranular pores connected by smaller throats provide the ions with important alternative paths to the intergranular conduits connected by larger throats. Therefore, for a consolidated rock such as Berea sandstone, we find no unique relationship between effective formation factor and electrolyte saturation, nor do we find a unique definition of the Archie saturation exponent, n, for the full range of saturation. Finally, the Archie saturation exponent

n is found to vary from approximately 3 when connected grain-contact pore space (i.e., thin sheets and micropores) and intergranular pores connected by smaller throats are filled with hydrocarbon paraffin to approximately 5 when intergranular conduits connected by larger throats are filled with hydrocarbon paraffin, with a critical saturation (S_{crit}) of 0.7.

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Predicting the Capillary Pressure of Berea Sandstone from Microgeometry

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The macroscopic transport properties of porous (and fractured) media depend sensitively upon processes at the pore level, which are controlled principally by the geometry and connectivity of the pore space. In addition, the microphysical, microchemical, and microbiological processes at the pore level affect both the hydraulic and the electric properties of porous materials. Consequently, there is a need for a basic understanding of how pore morphology and other related factors can be used to predict single and multiphase physical properties of porous media, such as intrinsic permeability, relative permeability, and capillary pressure. Visual observation and analysis of Berea sandstone complete pore structure by means of pore casts reveal that the rock pore space is composed of grain-contact pore space (i.e., thin sheets and micropores) connected to larger intergranular pore segments and pore throats, with

the whole structure arranged in a three-dimensional irregular network of irregularly shaped pores (Schlueter et al., 1992a).

In our study, we have attempted to understand, through analysis and experiment, how the relationship between capillary pressure and saturation is controlled by the rock pore structure and the distribution of wetting and nonwetting phases in the pore space. For this purpose, we have made analytical calculations of capillary pressure on the basis of pore microgeometry. As a zero-order approximation, we have idealized the porous medium as consisting of an assembly of parallel capillaries of arbitrary cross sections. The mathematical expression for capillary pressure as a function of saturation depends on the distribution of pore hydraulic radii and the area-perimeter power-law relationship of pores (Schlueter et al., 1992b). Two-dimensional scanning electron