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Micro-CT Tomography Assessment of Formation Factor from Cottesloe Clay-Sand Mixture of Perth Basin, Western Australia

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Abstract:

Electrical properties of rocks are important parameters for well-log and reservoir interpretation. Laboratory measurements of such properties are time-consuming, difficult, if not impossible in some cases. In this study, I developed a comprehensive and robust workflow with clay-sand from Cottesloe beach of the Perth Basin. Electrical conductivities were first carefully measured in the laboratory using a rectangular two-copper-electrode system. A porosity of 0.39 was achieved by compacting the samples in a way that reproduces natural packing of sand. 3D micro-computed tomography images of each sand sample were acquired. Image processing was done with Avizo fire using global thresholding method and up to 78 sub-samples of sizes from $(200)^3$ $(350)^3$ $(500)^3$ and $(700)^3$ voxels. In this research work 1 voxel is equal to $(2.6\mu\text{m})^3$. After segmentation, the images were used to compute the effective electrical conductivity of the sub-cubes using a Finite Element conductivity code (Free software from NIST; Garboczi, 1998) as well as formation factor. The clay-sand sample is in good agreement with the laboratory measurements and computation from digital cores was found, as long as the sub-cube size REV is reached that is between 8 and 11 grains.

Keywords: Cottesloe Clay, natural packing, NIST, REV

1. Introduction

Electrical properties of rocks are important parameters for well-log and reservoir interpretation. Laboratory measurements of such properties are time-consuming, difficult, and are impossible in some cases. Being able to compute them from 3D images of small samples will allow generating massive data in a short time, opening new avenues in applied and fundamental science. The electrical properties of sand depend on its composition, microstructure and interfacial effects; this includes the bulk properties of the sand and the geometry of the constituents of the sand. The electrical conductivity of saturated sand results from the conduction through the bulk solution occupying the pores as well as the conduction occurring at the fluid grain interface. The three separate phases found in a porous medium are the grains, the fluid-filled pores and the interfacial region that is in most cases not considered. The fluid-mineral interaction is an interfacial effect which is particularly important for electrical properties of clayey samples (Olhoeft et al., 1987). Surface electrical conductivity in rocks is an important parameter commonly used in characterizing hydrocarbon reservoirs Waxman and Smits (1968). The effect of the specific surface conduction can be from contributions of (a) conduction within the diffuse electrical layer, which is small but cannot be neglected in the total specific surface conductance; (b) conduction in the Stern layer, which has been shown to vary significantly with the salinity of the pore fluid at low salinities; (c) a system operating directly on the mineral surface, independent of salinity. The macroscopic conductivity of a saturated porous medium with insulating grains results from two mechanisms: (i) bulk conduction, which corresponds to electron migration of the ions in the interconnected pore space, and (ii) surface conduction, which corresponds to electrical conduction near the fluid-grain interface e.g. (Pride, 1994; Revil and Glover, 1997). The surface of a grain, when it is in contact with an electrolyte, is typically charged with specific conductance of quartz ($8.9 \times 10^{-9} \text{ S}$) while the conductivity of clay is ($2.5 \times 10^{-9} \text{ S}$). The effect of surface conductivity was studied by researchers like Archie (1942); Waxman and Smits (1968); Johnson and Sen (1988) and Knight and Nur (1987). The existence of a rough interface can produce a local reduction of the strength of the electric field in the vicinity of the mineral surface which modifies the contribution of the interfaces to the total electrical conductivity (Schwartz et al., 1989).

When an electrolyte is exposed to a solid mineral surface, either there will be an electrochemical reaction where a double-layer is formed. When the mineral grain has an unbalanced structural charge, a layer of excess ions develops in response to the local potential (clay particles for instance) that are negatively charged, hence, in solution, they attract an excess of cations. This creates what is referred to as the "double layer", composed of the fixed negative charge on the clay particle and the mobile positive charge in a thin solution layer adjacent to the interface (as shown in figure 1) far away from the surface, the densities of both positive and negative ions are equal.

Figure 1: The Electrical Double Layer (A) Gouy-Chapman Model
(B) Stern Model After Ruffet Et Al. (1995)

Conductivity will depend on frequency when there is diffusion, or when the diffusion occurs through a double layer, and the dependency will have an effect only at very high frequencies since the time constant to establish the double layer is very short unless the surface is rough.

The second conductive component was first detected and described for sandy clay by Patnode and Wyllie (1950). They found that Archie's relationships did not apply to sandy clay; it was apparent that the current was carried through a medium other than the saturating solution. They called this additional conduction "conductive solids" which include wet clay components in the form of disseminated particles. Clay and grain surface phenomena create a "double layer or interface conductivity" and electrical double-layer (Stern model). The two components are combined into a parallel conductor system Figure 2.

$$\sigma_{shalyrock} = \sigma_{electrolytic} + \sigma_{clay} = \frac{\sigma_{water}}{F} + \sigma_{clay} \quad 1$$

Figure 2: Schematic Representation of the Electrical Double Layer Situated at the Mineral Surface (Revil And Glover, 1997)

When thermodynamic conditions are different from the electrical diffuse layer (EDL) condition, the mineral surface S gets an excess of charge through ionization reactions. This excess charge is balanced by mobile ions in an electrical diffuse layer. The parameter σ_c is the effective electrical conductivity of a representative elementary volume (R.E.V.), whereas σ_f and σ_m are the free electrolyte and matrix conductivities respectively. The counterion of the electrical diffuse layer are maintained at some distance from the mineral surface by the water adsorbed on the surface forming the

“Stern layer”, and a hydration shell around each the cation. The Stern layer and the diffuse layer comprise the so-called “double layer”. (Figure 2).

This double layer formation is due to the presence of clay (e.g. Kaolinite in this research work) and is dependent on the cation exchange capacity (CEC), the cation CEC is the total capacity of a porous media to hold exchangeable cations. CEC is an inherent porous media characteristic and is difficult to alter significantly and sands with higher clay fraction tend to have a higher CEC.

The presence of conducting minerals example kaolinite changes the pore surface conduction, producing a second conducting component and therefore, Archie's equation must be modified as in Equation 1 (e.g. Kaolinite in figure 3 and figure 4).

Figure 3: 2D Schematic Representation of Structure and Composition of Kaolinite Clay Minerals

Figure 4: 3dschematic Representation of Structure and Composition of Kaolinite Clay Minerals

Clay minerals have negatively charged sites on their surfaces that absorb and hold positively charged ions (cations) by electrostatic force. When an atom or molecule loses or gains one or more electrons, then it turns into either a positive (cation) or negative (anion) electric charge respectively. The common cations found in solutions are hydrogen (H^+) and sodium (Na^+) as in the case of my research work, but other ones include potassium (K^+), calcium (Ca^{2+}), magnesium (Mg^{2+}), ammonium (NH_4^+) and aluminium (Al^{3+}).

There are many models of the physical explanation of clay conductivity based on the double layer proposed by researchers like Winsauer and McCardell (1953), Guyed and Wichmann (1971), and De Witte (1955). But the two most popular models are the ones proposed by Waxman and Smits (1968) and Bulling and Breyer (1989).

Waxman and Smits (1968) published their theory and model for sandy clay. In this model, the “electrical current associated with clays travels along the same tortuous paths as current attributed to ions in pore water”. This results in relationship with unique formation factor F

$$\sigma_0 = \frac{1}{F} \cdot (\sigma_w + B \cdot Q_v) \quad 2.$$

Or in terms of resistivities

$$\rho_0 = F \cdot \rho_w \cdot (1 + B \cdot Q_v \cdot \rho_w)^{-1} \quad 3.$$

The empirical parameter (counterion mobility) after Waxman and Thomas (1974) describes slight interface conductivity dependence on salinity.

$$B = 3.83 * \left[1 - 0.83 * \exp\left(\frac{-0.5}{\rho_w}\right) \right] \quad 4.$$

Clay parameter Q_v , is related to the cation exchange capacity (CEC) by

$$Q_v = \frac{1-\phi}{\phi} \cdot \frac{d_s}{100} \cdot CEC \quad 5.$$

where d_s are the solid grain density (in g/cm³) and ϕ porosity; CEC is in milliequivalents per 100g of sand and measured in the laboratory. The CEC is the ability of sands to hold positively charged ions or the total capacity of the sands to hold exchangeable cations; different materials have different cation exchange capacities. Table 1 gives the cation exchange capacity of different clays after McNeill (1980) while table 2 show the cation exchange capacity of kaolinite (the concern of this research work) after Keller and Frischknecht (1966) as shown below.

Soil type	Organic matter in %	Clay in % by weight	CEC in meq/100g
Sand	1.7	7.0	6.3
Sandy loam	3.2	13.2	13.7
Loam	4.9	16.8	20.2
Silt loam	5.4	18.4	24.0
Clay loam	5.5	31.2	27.2

Table 1: Cation Exchange Capacity (CEC) and Colloid Content of Five Soils of Different Textures After: McNeill (1980)

Clay type	Kaolinite	Mica	Chlorite	Montmorillonite	Vermiculite
Cation exchange capacity (meq/100g)	3-15	10-40	10-40	80-150	100-260

Table 2: Table of Cation Exchange Capacity of Various Clays: Keller and Frischknecht (1966)

Bulling and Breyer (1989) showed that in dispersed clay, electric current flowed through a mixture of pore water and dispersed clay in pores as:

$$F_{im} = \frac{a}{\phi_{im}^m} \quad 6.$$

and the specific resistivity of a water clay mixture parallel model (weighted arithmetic mean for conductivity) is applied:

$$\rho_{slurry} = S_{sim} \cdot \left(\frac{S_{sim} - q}{\rho_w} \right) + \left(\frac{q}{\rho_{clay}} \right)^{-1} \quad 7.$$

where ρ_w is formation water resistivity and ρ_{clay} resistivity of the dispersed clay.

ϕ_{im} = intermatrix porosity, which includes the space, occupied by fluids and dispersed clay.

S_{im} = the fraction of the intermatrix porosity occupied by the formation-water, dispersed clay mixture.

q = the fraction of intermatrix porosity occupied by the dispersed clay.

Then the conductivity of the dispersed clay results is

$$\sigma_{dis} = \rho_{dis}^{-1} = (\rho_{slurry} \cdot F_{im})^{-1} = \frac{\phi_{im}^m}{a \cdot S_{im}} \cdot \left(\frac{S_{im} - q}{\rho_w} + \frac{q}{\rho_{clay}} \right) \quad 8.$$

Cation exchange capacity is a typical property of clays and relates to the clay mineral structure. Because of the geometric nature of these fundamental structural units, structures are sheet-like, one type consists of octahedral units of oxygen or hydroxyl around a central atom (usually aluminium), and the other type is tetrahedral unit composed of a central silicon atom surrounded by oxygen (Figures 3 and 4). The fluid-mineral interaction is an interfacial effect which is principally significant for the electrical properties of clayey sand samples Olhoeft et al. (1987).

The two phenomena that relate to the physical properties of clay are:

- The presence of water trapped between "plates" with its influence on conductivity and porosity measurement are not part of effective porosity.
- The presence of a strong negative surface charge with the ability to absorb ions (e.g. radioactive ions) creates a double layer of pore fluid.

The capacity of the clay minerals to form an electric double layer is the basis for cation exchange phenomena. During the mixing of saline water and clay, positive charges (cations) are absorbed on the clay's surface. Cations (Ca, Mg, H, K, and Na) are loosely held on the surface and can subsequently be exchanged for other cations or essentially go into solution should the clay mix with water. For this reason, they are called "exchangeable ions" and the cation exchange capacity is a

measure of the number of cations that are required to neutralize the clay particle as a whole; that is, the weight of ions in milliequivalent absorbed per 100 grams of clay.

When evaluating adsorption, the specific surface area of sand grains is usually not of much interest compared to that of clays contained in the sand. Table 3 gives the range of specific areas expected from clays by Corey (1994). The effect of the surface area is, therefore, negligible in my research work because I used kaolinite that has the least surface area effect as can be seen from table 3 below.

Clay type	Area m ² /gram
Kaolinite	45
Illite	175
Montmorillonite	800

Table 3: Specific Surface Area of Different Clay Types After Corey (1994)

In clay-sand there is an additional charge carrier in the fluid phase, the Kaolinite clay added was 3% by weight of the sand sample, it is dried in an oven for seventy-two (72) hours at a temperature of 105°C, and then the dry clay was thoroughly mixed by hand for about forty (40) minutes. Kaolinite clay contains charged impurities like Al³⁺ substituting Si⁴⁺; the charge deficiency is compensated for cation adsorption on the mineral surface. This adsorption creates an electrical double layer near the mineral surface as shown in Figure 2, with the concentration of ions in solution extending 30 to 80Å from the surface. Surface conduction is due to these ions in the diffuse double layer and is localized near the clay surface.

2. Materials and Method

The samples investigated in this research work are sand samples collected from the coastal margin of the Perth basin, Western Australia. The Perth Basin is an elongate, North-South trending trough underlying approximately 100,000sqkm of the Western Australian margin. Sediments were shed from the adjacent Yilgarn block. The Yarragadee and Leederville sandstone-formations are intercalated with the Tamale limestone that forms the Carbonates at the Upper Cretaceous. One sample was collected from Cottesloe beach (31°59'40.62 S, 115°45'03.70 E). The sample is composed of quartz and carbonate. Grain size was determined by micro CT-image analysis and is between 17µm - 606µm (median 124.0µm).

Sand samples were thoroughly washed clean with tap water to remove any plants and grass debris. Loose moist clay-sand was then packed into the rectangular cell (figures 5 and 6) used to perform the electrical resistivity measurements, then forming an initially high-porosity loose random pack; decreasing porosity in subsequent experiments was achieved by shaking the cell and using tied sticks to compact the sand: this was done in a way to achieve a packing as close as possible as the one found in-situ.

Porosity was determined from the weights and densities of the sand grains and the known volumes of cells used in the experiment, as:

$$\Phi = [v_t - m/\rho]/v_t$$

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where Φ is porosity, v_t is the total volume of the cell, m is the average mass of the dry sand before and after the experiment and ρ is the density of the sand grains. Grain density was measured by pycnometry and found to be equal to 2.71 g/cm³.

Figure 5: Schematic Diagram of Rectangular Cell for the Experiment

Figure 6: Rectangular Cell for the Experiment

3. Result

3.1. Laboratory Result of Clay Sand

The graph of the electrical conductivity of the formation of the Cottesloe clay-sand against time presented in 8 show a very longer time of about 42 days to complete the experiment. The graph of the conductivity of formation against the conductivity of water in Figure 7 shows how the readings became unstable especially at higher salinities of 25mg/l and 35mg/l saline solutions. The value of the porosity and formation factor computed from the laboratory measurements is 0.39.

Figure 7: Cottesloe with 3% Clay (A) Conductivity of Formation against Time (B) Conductivity of Formation against Conductivity of Water

3.2. Micro CT-Scan Image Result of Clay Sample

The Cottesloe Beach clay-sand was prepared for imaging with micro-CT: loose, moist clay-sand was sand put in a cylindrical Pyrex glass tube of 6 mm in diameter and 6 cm in height. It was inserted into a core holder of a micro-tomograph and the sample was scanned with a 3D X-ray Microscope Versa XRM 500 Zeiss – XRadia as shown in (figure 8) using X-ray energy of 60keV, a current of 70.66 mA and energy of 5×10^6 Watts. The images were scanned using two different resolutions of $3.4348 \mu\text{m}$ with a total number of 2D images of 990. These images were acquired with exposure times of 9.51s with a total scanning time of 2 hrs 6 minutes and 2seconds. Nominal voxel sizes of $(3.4348 \mu\text{m})^3$ were achieved with source-to-sample and detector-to-sample distances of 11mm and 11mm for the images (figures 9 and 10). Initial cone-beam 3D image reconstruction was performed using initial software XM Reconstruction (XRadia).

Figure 8: Cylindrical Pyrex Glass and X-Ray Micro City Scan (Versa XRM 500)

Figure 9: Cottesloe Clay-Sand Sample

Figure 10: Adjusted and Filtered Micro CT-Scan Voxels of Cottesloe Clay-Sand Image

Figure 11: Threshold Image of the Clay-Sand Showing Pores (Black), Quartz (Blue), Carbonate (Grey) Clay (White)

After filtering and threshold, the clay-sand image was cropped into 10 cubes of 700^3 , 15 cubes of 500^3 , 25 cubes of 350^3 and 30 cubes of 200^3 as shown in figure 12.

Figure 12: Cropping of the Clay-Sand Image (A) 700^3 , 500^3 , 350^3 , 200^3 Cubes

Figure 13: Formation Factor Against Porosity of Cottesloe Beach Samples of Clay-Sand 7003, 5003 3503 And 2003 Cubes

Figure 14: Porosity against Cube Sizes of Cottesloe Clay-Sand

Figure 15: Porosity Against Cube Sizes of Cottesloe Clay-Sand

4. Grain Size Analysis

There are 1,604 grains in the Cottesloe Beach clay-sand sample with 401 grains of quartz, 437 grains of clay, 473 grains of carbonate and 293 pores. The smallest grain size found within the quartz particles in the Cottesloe clay sand sample has a diameter of $14.2\mu\text{m}$ with the largest grain having a diameter of $840.9\mu\text{m}$. The range of the smaller grains from $14.2\mu\text{m}$ to $29.8\mu\text{m}$ constitutes 15.9% of the quartz grains of the Cottesloe clay-sand sample while the largest grains ranging from $550.6\mu\text{m}$ to $840.9\mu\text{m}$ constitutes only 2.8% of the total quartz grains. The remaining quartz grains are of intermediary grain sizes. The smallest grain size found for clay in the Cottesloe clay sample is $15.6\mu\text{m}$ with the largest having a diameter of $375.2\mu\text{m}$. The range of smaller grains of clay from the Cottesloe clay-sand samples is $15.6\mu\text{m}$ to $36.0\mu\text{m}$ constituting 24.7% of the total Clay while the largest grains ranges from $199.5\mu\text{m}$ to $375.2\mu\text{m}$ and constitute 12.8% of the total clay grains. The smallest grain size found for Carbonate in the Cottesloe clay-sand sample is $14.5\mu\text{m}$ with the largest having a diameter of $519.3\mu\text{m}$. The range of smaller grains of Carbonate from the Cottesloe clay sample is $14.5\mu\text{m}$ to $50.0\mu\text{m}$, constituting 12.9% of the total Carbonate while the largest grain ranges from $298.0\mu\text{m}$ to $519.3\mu\text{m}$ and constitute 12.9% of the total carbonate grains. The pores in the Cottesloe clay sample have a least pore diameter of $8.2\mu\text{m}$ with the largest pore of $840\mu\text{m}$. The smaller pores, ranging from $8.2\mu\text{m}$ to $39.9\mu\text{m}$, constitute 26.3% of the total pore count while larger pores ranging from $499.3\mu\text{m}$ to $840.9\mu\text{m}$ constitute 27.6% of the total pore count Figure 14 and table 4.

Figure 16: Frequency Distribution of Components in the Cottesloe Clay-Sand
(A) Pore (B) Quartz (C) Clay and (D) Carbonate

Beach	Material	Range(μm)		Average(μm)
Cottesloe clay sand	Pore	Min	8.15	182.0
		Max	840.9	
	Quartz	Min	14.2	196.3
		Max	740.2	
	Clay	Min	15.6	68.1
		Max	375.2	
	Carbonate	Min	14.5	172.9
		Max	519.3	

Table 4: Summary of Equi-Diameter of materials

4.1. Computational Plots of the Electrical Potential Field after Finite Element Code

Using free codes from National Institute of Standards Technology (NIST), I used the output file from the conductivity calculation of all the 78 cubes (images shown in Figure) to plot the potential field variation of each cube. Shown below is an example of the result of the potential image (Figure 10) of the average conductivity.

Figure 17: Cottesloe with 3% Clay Showing the Components: Deep Blue (Pore); Sky Blue (Quartz); Maroon (Carbonate); and Yellow (Clay) of (A) (700)3 (B) (500)3 (C) (350)3 and (D) (200)3 Cubes

Figure 18: Cottesloe with 3% clay showing potential field variation of (a) (700)3 (b) (500)3 (c) (350)3 and (d) (200)3 cubes

Laboratory Results	Static cell	Porosity	0.39												
		F.F	6.844												
Micro-CT scan images	(700) ³ cubes	Porosity	0.405	0.405	0.405	0.405	0.405	0.404	0.404	0.404	0.404	0.404			
		F.F	4.269	4.272	4.272	4.279	4.284	4.285	4.302	4.302	4.310	4.310			
	(500) ³ cubes	Porosity	0.414	0.414	0.414	0.414	0.414	0.413	0.413	0.411	0.410	0.409	0.407	0.407	0.404
		F.F	4.090	4.107	4.107	4.132	4.187	4.212	4.221	4.260	4.265	4.265	4.299	4.299	4.318
		Porosity	0.404	0.402											
		F.F	4.329	4.339											
	(350) ³ cubes	Porosity	0.479	0.473	0.438	0.438	0.416	0.410	0.408	0.400	0.400	0.390	0.387	0.382	0.376
		F.F	3.902	3.922	4.032	4.051	4.231	4.355	4.407	4.426	4.429	4.441	4.498	4.516	4.546
		Porosity	0.371	0.370	0.367	0.362	0.361	0.360	0.359	0.351	0.349	0.338			
		F.F	4.600	4.623	4.743	4.781	4.799	4.851	4.945	5.083	5.324	5.394			
	(200) ³ Cubes	Porosity	0.661	0.630	0.567	0.515	0.497	0.437	0.430	0.422	0.412	0.411	0.382	0.378	0.373
		F.F	1.978	2.495	2.537	3.297	3.313	3.472	3.795	3.817	3.923	4.134	4.421	4.538	4.603
		Porosity	0.363	0.362	0.362	0.361	0.348	0.346	0.344	0.336	0.334	0.319	0.304	0.293	0.283
		F.F	4.695	4.996	5.106	5.246	5.517	5.671	5.631	5.635	5.715	5.717	5.902	5.934	6.268
		Porosity	0.275	0.262	0.252	0.248									
		F.F	6.691	6.796	6.946	7.497									

Table 5: General Table for the Cottesloe Clay Sand Mixture

Figure 19: Summary of Measurements from Laboratory and Micro Ct-Scan Images from Cottesloe Clay Sand

5. Discussion and Conclusion

The estimation of conductivity from the digital sample of clay-sand involves the three-phase segmentation of the micro-CT image of these mixtures. The over or under segmentation of these samples can over or under estimate the percentage of pores that can contribute to the electrical conductivity (porosity). The clay phase, as it has intrinsic porosity, in the grey scale images is an intermediate phase between solids and pores. Therefore, a simple threshold segmentation is not enough to segment the image correctly, therefore, global threshold was employed. After clay segmentation, the electrical conductivity of the water-saturated clay-sand mixture is estimated from the finite element code, by assuming an effective electrical conductivity. We can assume the clay has an effective conductivity, related to its intrinsic porosity, which is lower than the resolution of the micro-CT images.

This research work could address the issue of scaling, although results obtained from laboratory scale (cm) with that of micro-CT the images are not of the same scale, but both results follow a similar trend. Therefore, extrapolating it to field scale (m or even km) to follow the same trend is feasible. Computing for the formation factor of grains with intra-granular porosity (carbonates), as they are characteristic of Cottesloe Beaches. I was also able to alter the code to compute for the formation factor of clay-sand. I envisage that, computing the formation factor of more complex samples, such as sands with disseminated conductive grains (e.g. ores), could be achieved by accurate alteration of the NIST code. A robust method of obtaining the formation factor from CT-scan images that takes a shorter time (1 day) from that of laboratory measurements (that take a much longertime of 3-7 days for a clean sand to 30-65 days (in case of clay-sand) was established.

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