# LABORATORY INDUCED DAMAGE - A REVIEW OF THE PROBLEM

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ABSTRACT Laboratory data indicating possible formation damage can too often be a result of laboratory practices and be misinterpreted as a reservoir problem. All core handling, cleaning, drying, preparation and analysis procedures can, if incorrectly performed, be a source of laboratory induced damage, especially on core from formations with delicate mineralogy.

Fluid flow tests, in particular, are potentially damaging. For many years the industry standard for liquid permeability and unsteady-state relative permeability testing has required an end-point flow rate of 6 cm³.min⁻¹. When this is scaled to field conditions it greatly exceeds flow velocities usually encountered, typically 166 ft.day⁻¹ through a 1.5" sample with 15% porosity. These excessive laboratory flow rates can cause movement of fines, or non-Darcy flow which provides data of limited value. Flow pressures required to achieve a flow rate of 6 cm³.min⁻¹ can be very high, requiring, in turn, even higher confining pressures which may cause stress damage to the core sample. Analysis of flow pressure variation during a constant flow rate test can indicate if permeability damage has occurred.

Laboratory techniques exist to avoid problems. Detailed sample screening prior to any other analysis is invaluable, to identify the sample mineralogy and mineral morphology. Flow parameters are established by determining critical velocity, the velocity above which fines become mobile, or Darcy flow is exceeded. With critical velocity information all subsequent flow tests can be designed to yield data which is representative of reservoir behaviour.

### INTRODUCTION

Laboratory induced damage can be defined as the physical and chemical alteration of core samples during analysis. This alteration can be caused by the preparation and/or test procedures used in the laboratory. The result is data that is not representative of the reservoir under evaluation.

The problem is widely recognised and published research has identified many laboratory processes and techniques that can be a cause of damage. This damage is related to the formation characteristics and the mineralogical complexity.

Engineers, petrophysicists and geologists, who have to plan the optimum core analysis programme are often faced with the difficult task of deciding on what handling, preparation and test procedures should be used.

This scope of this paper is to highlight areas where laboratory procedures may be a cause of sample damage, and as a result yield data that is unrepresentative or erroneous. As a solution to the problem, an approach that relates understanding of the test limitations to the mineralogy and the mineral morphology is required.

# CORE HANDLING/SAMPLE PREPARATION

All core handling, cleaning, drying and preparation procedures can, if incorrectly performed, be a source of laboratory induced damage. Wellsite handling and preservation are also potential problem areas and should be recognised as such. The purpose of this paper, however, is to concentrate on laboratory practices only.

When a core arrives in the laboratory it is often the case that the complete analyses requirements have not been decided or, if they have, not communicated to, or requested by, the service laboratory. This situation poses the service laboratory with the dilemma of balancing the normal priority of rapid turnaround versus planning and implementing the optimum core handling and sample preparation programme. In many cases the only requirement that has been communicated to the service company is that of "hotshot data" within 24 hours of receipt of core in the laboratory. To meet this requirement in many cases involves proceeding with handling and core preparation without knowing if they are compatible with the rock type.

# Plug Drilling

Use of a non-compatible plug drilling fluid can cause damage to the plug sample and damage to the remainder of the core in contact with the plugging lubricant. This incompatibility can be a fluid/rock reaction or a fluid/fluid reaction or both. The damage mechanisms are discussed later.

# Sample Cleaning

Cleaning of the samples can cause opportunities for laboratory induced damage. Numerous methods for core and plug cleaning are used within the industry, examples of which are described as follows.

Soxhlet cleaning is the most commonly used plug cleaning system. Various solvents or solvent mixtures can be used, with various boiling points and varying degrees of cleaning effectiveness. Methanol and toluene are the solvents most routinely used to extract salt and hydrocarbons respectively. The most commonly recognised problem with these solvents is that of the relatively high boiling points causing clay dehydration. Another is naturally occurring halite being removed from the rock matrix causing subsequently erroneous pore volume determination. Toluene is not an effective solvent for removing heavy hydrocarbons. The result is that heavy ends are deposited onto the grain surfaces rendering the sample oil wet or partially oil wet, so subsequent fluid flow tests may yield erroneous data if the formation is in fact water wet.

If a formation is recognised as having delicate mineral morphology, low flow rate flush cleaning is normally recommended. The difficulty is deciding which flow rate to use and how to minimise the volume throughput and yet effectively clean the samples.

# Drying

Hot oven (105 degrees Celsius) drying of samples to remove the solvents prior to analysis is a major source of induced damage. Problems occur as the immiscible air-solvent interface passes through the sample. Studies have shown that in cores containing illite the interfacial tension at the gas liquid interface passing through the sample causes collapse of the fibrous structure. This collapse and matting of the illite on to the pore walls causes subsequent permeabilities to be erroneously high. Critical point drying

procedures preserve the delicate illite structure, and subsequent permeabilities are representative.

High temperature drying can also cause clays to become brittle and flaking can occur. These broken particles can become mobile during subsequent flow tests and cause pore throat plugging and a reduction in permeability.

### **FLOW TESTS**

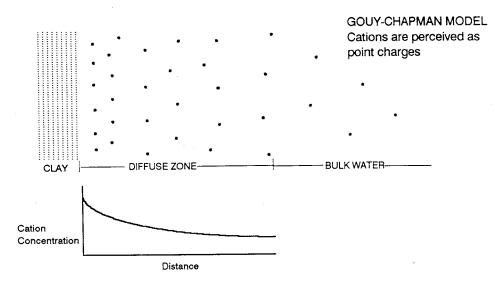
# Incompatible Saturant or Flowing Brine

Formation brine in a reservoir is in chemical equilibrium with the minerals it surrounds. Water sensitive minerals are colloidal process controlled and are therefore sensitive to salinity, pH and/or cation composition changes.

Monaghan et al (1959) concluded that salinity is important in controlling permeability reduction due to clay alteration. Even a 1% calcium chloride solution is effective in preventing damage. Mungan (1965) noted pore throat blockage by fine particles can be caused by salinity change and/or pH change. Jones (1964) also noted that small proportions of divalent cations, such as calcium or magnesium, in the saturant brine cause resistance to clay dispersion. He also noted that a slow gradual change from high to low salinity is less damaging than a sudden change. Hewitt's (1963) experimental work demonstrated the two main permeability reduction mechanisms, particle movement and clay swelling. He also recognised the value of mineral identification by X-ray diffraction.

More recent work by Kia et al (1987) reported the effect of salt composition of injected fluids on water sensitivity. The presence of calcium ions in a solution containing sodium ions reduces permeability decline typically found when fresh water contacts a rock with prior exposure to sodium ions. Calcium ions are selectively adsorbed onto the clay particle surfaces, and a critical calcium surface coverage of 75% is required to eliminate formation damage. This effect was attributed to double layer interactions between clay particle surfaces and pore walls.

The double layer theory relates interactions among clay minerals, interlayer cations (in the clay structure), pore water and pore water cations. The negative charges on clay surfaces are balanced by cations in solution in the pore water, and these cations are in greater concentration nearer the clay surface (Figure 1). Cations in the



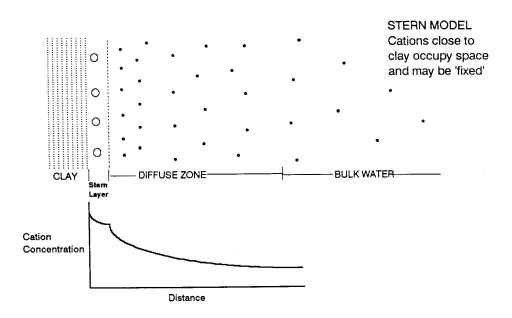


FIGURE 1 The Double Layer Theory

diffuse layer are hydrated, and this association of cation and polar water molecule results in marked increases in the size of the "cation". The largest hydrated cations are +2 e.g.  $\text{Ca}^{2+}$  and, due to their high surface charge density, are preferentially adsorbed to the clay surface. For any valence system, however, the smallest cations e.g.  $\text{K}^{+}$  are preferentially adsorbed.

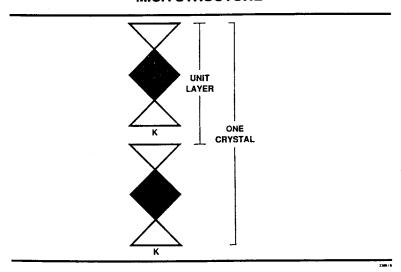
Swelling of clay minerals occurs when water is added to the interlayer spaces (Figure 2). Initially the water hydrates the interlayer cations, but after four water molecules have been added the clay may disperse if electrostatic forces of repulsion become significant.

# SMECTITE STRUCTURE UNIT LAYER ONE CRYSTAL T-H<sub>2</sub>O & Mg, Na, Ca

FIGURE 2 Swelling clay structure.

Reed (1977) showed that other minerals such as mica and carbonate cements could be disturbed by contact with incompatible water. Carbonate cements could be dissolved to free particles to move and block pore throats. Mica could be altered to easily dispersable particles by removal of potassium from interlayer sites. Even when flowing high concentration polyvalent salt solutions, devoid of potassium, potassium is extracted from the mica (Figure 3). Exchange of potassium for larger hydrated ions such as calcium

# **MICA STRUCTURE**



# **EFFECTS OF MICA ALTERATION**

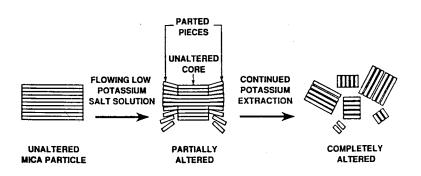


FIGURE 3 Alteration of mica.

or sodium in the interlayer spaces causes expansion and stress within the mineral structure. This leads to small expanded particles breaking off and dispersing in the flowing fluid, and then plugging pore throats reducing permeability.

### **Excessive Flow Rate/Pressures**

Traditionally the service industry standard for liquid permeability and unsteady state relative permeability testing has required an end-point flow rate of 6 cm<sup>3</sup>min<sup>-1</sup>. When scaled to field conditions it greatly exceeds flow velocities usually encountered, typically 166 ft.day<sup>-1</sup> through a 1.5" diameter sample with 15% porosity.

This very high flow rate has been used to minimise capillary end effects. Many authors e.g. Kyte and Rapoport (1958), Richardson and Perkins (1957), Rapoport and Leas (1953), Perkins (1957) investigated this. The general conclusion was that capillary hold up of the wetting phase can be minimised by using high flow rates or long core columns.

However, the high flow rate became normal for all types of liquid permeability tests. Even in tests where only one liquid is flowing, where there are no capillary end effects, the high flow rate was used. High flow rates in laboratory tests can cause reduced permeability due to either turbulent flow, movement of fines or excessive confining pressure at the downstream end of the sample.

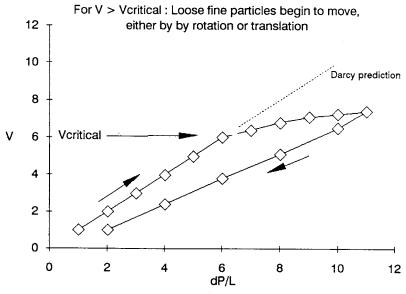


FIGURE 4 Rate versus pressure differential

Turbulent flow or non Darcy flow causes a reduction in the kinetic energy of the flowing fluid. Therefore an increase in pressure differential is required to flow the fluid at the required rate, leading to a calculated decrease in permeability. This can be investigated in the laboratory by flowing fluid at increasing rates and plotting rate against pressure differential per unit length (Figure 4).

Almost all hydrocarbon bearing sands and sandstones contain minerals (clays and silts) which are potential sources of mobile fine particles. Fines migration damage may be caused by a variety of fines including clays, amorphous silica, quartz, feldspars, mica and carbonates.

The mechanism of fines dispersion and movement due to water composition (salinity, salinity change, pH and ionic strength) has been reviewed above. However, fines migration is primarily controlled by hydrodynamic drag. Fine particles are either cemented to the host grain or loosely attached within the pore network. Authigenic kaolinite and illite are very susceptible to migration due to their very large surface area to volume ratios and their loose attachment to pore walls.

Loosely attached fines not cemented to grains can move if viscous and interfacial forces promote movement. Even fines which are well cemented to the matrix will move if the hydrodynamic drag caused by moving fluid exceeds the intrinsic bond strength holding the fines particles to each other or to the host matrix.

Permeability reduction occurs either when the fine particles arrive and block a pore throat which is smaller in size than the individual particles, or when several small fine particles reach a larger pore throat at the same time and thus bridge the throat causing blockage.

Gruesbeck and Collins (1982) demonstrated that fines movement can cause permeability impairment, that fines are not restricted to clay minerals and that a minimum interstitial fluid velocity is required for fines movement. Gabriel and Inamdar (1983) reported that both mechanical and chemical mechanisms can cause mobilisation of fines. Egbogah (1984) defined a laboratory procedure to determine a critical flow rate above which fines movement becomes a pore plugging problem, and that this rate is unique to every formation.

Muecke (1979) showed that fine particles move only if the phase that wets them is moving. Therefore, fine particles which are waterwet may move only when water saturation exceeds a critical level during a water displacing oil flood.

Amaefule et al (1987) used critical velocity tests to evaluate the maximum flow rate at which to perform relative permeability. Critical velocity tests were performed with different fluids (oil and water) at a variety of saturation levels. Flow rates were plotted against differential pressure per unit length. At some high rate the pressure drop became higher than Darcy's Law would predict. This is indicative of permeability reduction. Further reduction in flow rate would show the pressure drop return to the straight line relationship if turbulent flow was the mechanism, but would not follow the previous line if fines movement was the mechanism (Figure 4).

A plot of permeability versus interstitial velocity indicates the critical velocity. In addition, pH monitoring of the effluent brine is an indication of chemical change within the sample (Figure 5).

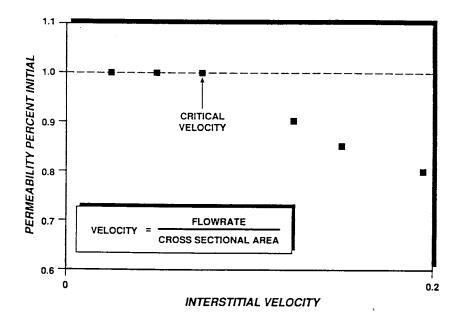


FIGURE 5 Critical velocity.

Another effect of the traditional requirement for high flow rates in laboratory liquid permeability tests is the requirement for high confining pressure. In low permeability cores a high pressure differential is required to achieve a flow rate of 6 cm³min⁻¹. Therefore, a confining sleeve pressure in excess of this pressure differential is required to avoid liquid bypass. However, as the pressure of flowing fluid decrease towards the downstream end of the sample, the net confining stress correspondingly increases (Figure 6). This sometimes results in a confining stress even greater than reservoir stress being applied to core samples. This can cause pore restriction and permeability damage, even mechanical failure, and invalid results.

# Minimising Laboratory Induced Damage

The above is a summary of the steps in the core analysis process where laboratory induced damage can occur. The possibilities are numerous and varied. The extent of the damage induced is directly related to the interaction between the core handling, preparation, test process and the rock mineralogy and morphology. The damage can be a single isolated process or a complex chain of processes compounding the problem at each stage of the analysis.

To yield laboratory data which is most representative of reservoir behaviour requires an approach that plans the core handling and analysis programme according to the mineralogy and more specifically the chemical compositions of the rock. The reactions that cause damage are both physical and chemical, the complexities of which have to be recognised by the core analyst when planning an analysis programme. This planning must happen prior to commencing sampling the core in the laboratory, and should involve detailed sample screening.

Valuable tools are available to the core analyst for detailed sample screening, e.g. X-ray Diffraction and Infrared Spectroscopy to aid in whole rock and clay mineral identification. Infrared spectroscopy is particularly useful due to its speed (20 minutes preparation and analysis time per sample). This allows the analyst to have a complete mineral make-up within several hours of receiving the core in the laboratory. During the time it takes to run the spectral core gamma, mineral and clay type information is available to aid in deciding the plugging fluid to be used to cut the samples.

Scanning Electron Microscopy gives the analyst a means to visually examine the sample in detail. It gives information as to the morphology and distribution of potentially problematic minerals and clays. With this information an analysis programme can be designed to minimise the chances of laboratory induced damage.

A synthetic brine made in the laboratory according to a detailed chemical analysis of the formation brine is the best saturant. If a detailed analysis of the formation brine is not available, then a brine containing damage inhibiting ions, such as calcium and potassium for dirty sandstone formations, should be used.

Critical velocity tests should be performed in order to evaluate the maximum flow rate above which all further flow tests should not be performed.

# VARIABLE CONFINING PRESSURE

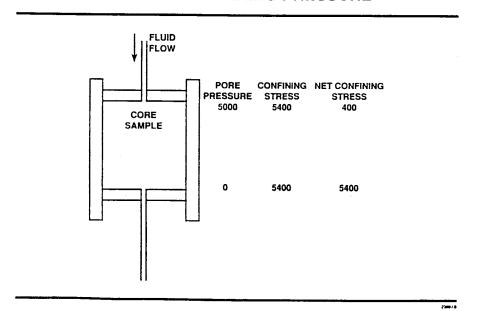


FIGURE 6

### CONCLUSIONS

Laboratory induced damage is a major problem that influences the validity of core analysis data interpretation. The opportunities for damage to occur are numerous and varied. Laboratory techniques exist to avoid problems but can only be used with a complete understanding of the mineralogy of the test sample. Detailed sample screening prior to any core handling or analysis is the only way to ensure that the subsequent data will be representative.

Use of compatible brines and critical velocity tests can increase the value of all further test results.

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