CORE SAMPLE PREPARATION – AN INSIGHT IN TO NEW PROCEDURES

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ABSTRACT

This paper aims to challenge many of the traditional methods employed in core analysis to prepare core plug samples for testing. Studies over many years have revealed that different lithologies can be altered in different ways during plug cutting, cleaning and drying.

Core plug samples for many core analysis measurements are removed from core and then trimmed to create right cylinders. The cutting and trimming of core plug samples can affect subsequent petrophysical measurements and some procedures will be proposed in the paper derived from analysis of many different rock types.

For many core analysis measurements, samples are cleaned of pore fluids and contaminants and then either measured in a dry state or with partial or full resaturation. Cleaning and drying of rock samples can be achieved using many different techniques. These techniques will be briefly reviewed and some new evidence presented which demonstrates severe damage to different rock types caused by some commonly used cleaning techniques. The development of a new modified Soxhlet cleaning technique will be presented formally for the first time.

This paper will present laboratory data, supported by SEM analysis which clearly illustrates the sensitivity of many different rock types to preparation techniques. Opinions, strongly held and based on a weight of evidence will be presented and we expect the paper to add to the already lively debate which exists on these topics.

INTRODUCTION

Core testing has many uses and applications in the oil industry. Reservoir capacity and performance can be predicted and understood using core testing. Well performance, development strategy and reservoir potential can also be examined. All core testing should endeavour to measure real rock properties and minimise the impact of testing equipment and core preparation procedures. Two factors which have been found to have significant impact on some core measurements and which have not been satisfactorily addressed previously are explored in this paper.

When core plugs are removed from core, they are usually cut as long cylinders and then trimmed to right cylinders of appropriate length. Cutting the core and trimming creates rock flour or “cuttings” some of which are usually deposited onto the core plug surface.

This layer of debris can affect all measurements made on the core plug sample. Various methods for removal of this rock flour have been proposed in the past. These include brushing dry or under liquid, blowing clear with air or nitrogen and trimming whilst flowing fluid out of the sample through the end faces. Other techniques which have been used include "sellotape" peel or the use of a vacuum to remove rock flour. Brushing can simply redistribute the flour and in some cases force it deeper in to the pores. Blowing with gas can also force the rock flour deeper in to the pores. Trimming in a confining device with flow can be laborious and can also promote fines migration. This paper proposes a simple technique which effectively removes rock flour and does not have any obvious disadvantages. This technique has been in regular use for more than ten years and has been very successful in ensuring accurate subsequent core measurements.

The majority of reservoir rock samples available for testing are either poorly preserved or not preserved. To proceed for testing they need to be cleaned of residual solids and fluids. There are many different methods for cleaning samples and the selection of the optimum method can have a profound effect on all results obtained from subsequent tests. One popular method for cleaning is to flow fluids or combinations of fluids through the plug samples in a core holder. Amongst the potential problems with this method are: potential for fines migration to occur prior to the real laboratory testing; poor cleaning efficiency – inefficient sweep of all pores; difficulty in achieving required saturation with subsequent fluids. Hot Soxhlet solvent extraction can also be used to rapidly clean samples. This technique is now largely discredited due to severe alteration to delicate minerals which can occur. The photographs presented in figures 4 and 5 are adequate evidence to condemn this technique where important decisions are to made on the basis of core testing. We believe that the best cleaning/drying methods are cold static cleaning and warm constant immersion cleaning. Cold static cleaning is normally prohibitively time consuming but warm constant immersion achieves good results in reasonable time. Samples are dried by low temperature oven drying to retain the intrinsic structures within the pore space. Cornwall[1] previously outlined how the continuous immersion technique can be used to control the rate and aggression of the cleaning process. The method described in this paper can be applied to all core samples in order to improve the quality of the cleaning process.

LABORATORY PROCEDURES

Acetate Peel To Remove Rock Flour

The method used for the removal of rock fines from the plug end faces is an adaptation of the stained acetate peel technique described by Katz and Friedman[2]. Although this technique was first used to produce rapid stained peels of limestones as a representative thin section, by adapting the method to that described below, it has proved to be very successful in removing the bulk of the rock flour from the plug end faces of sandstone and carbonate rock types.

A square section of acetate sheet approximately twice the size of the plug end face diameter is submerged completely in acetone solvent for up to 1 minute until it becomes jelly-like. This is then attached to a dry acetate sheet square and as soon as the jelly

acetate is attached to the dry acetate sheet, the wet surface is applied to the plug face and rubbed gently until no air bubbles are observed. The plug plus acetate sheet is then left to dry out at room conditions overnight. The acetate/acetone is allowed to harden or set. The acetate sheet should then be carefully peeled off the end face of the plug, which should now appear cleaner. A microscope can be used at this stage to ensure that the rock flour has been removed successfully.

The acetate peel technique is used to remove rock flour from flat end faces and curved surfaces of plugs and whole cores during cleaning. It can also be used to remove the contaminating layer of debris from preserved or fully saturated samples although this requires a small modification to the technique.

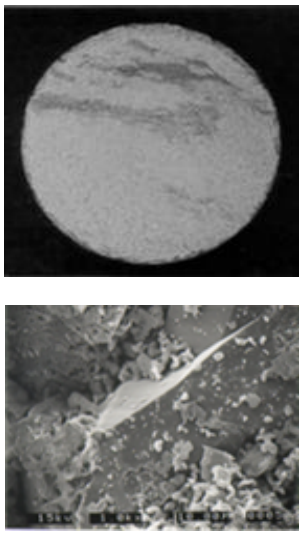

Figure 1. The upper photograph shows the plug face with no prior treatment. Immediately after the sawing and removal of the plug end trims. Rock fines occur in patches over the face producing a hazy effect. The lower photograph shows that few pores remain open (black patches), most being blocked by loose rock fines.

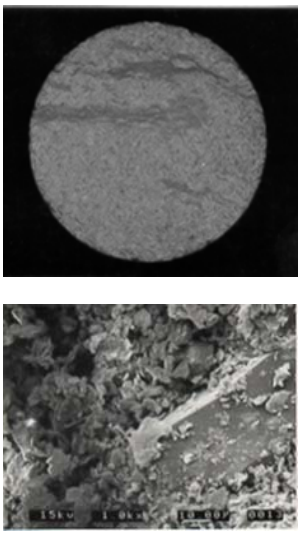

Figure 2. The upper photograph shows the plug face after being brushed to remove loose rock fines and appears cleaner than the untreated face shown in Figure 1. S.E.M. however, shows that all pores now contain rock fines (lower photograph). The brushing appears to have forced rock fines into pores previously open in the untreated sample.

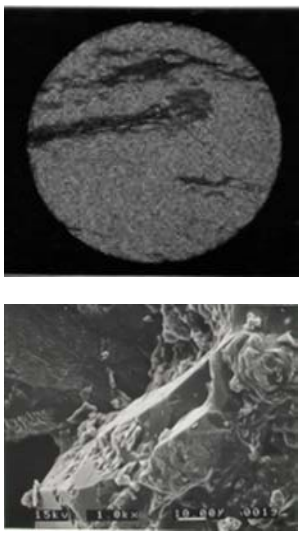

Figure 3. The upper photograph shows the plug face after being acetate peeled, to remove rock fines from the pores. When compared to Figures 1 and 2 the sample appears essentially free of rock fines. The lower S.E.M. photograph confirms that the pores are essentially free of rock fines.

The visual evidence presented in Figures 1-3 shows the removal of rock flour creates a fresh uncontaminated rock surface. Petrophysical measurements in the laboratory have confirmed that in some cases there is a direct impact on the absolute value of permeability measured. The greatest concern is for advanced testing with liquids such as relative permeability or formation damage testing. The data presented in table 1 shows that even air permeability can be affected by end face cleaning. Three lithologies or rock types are presented. In each case brushing the end face causes a small alteration in air permeability but in the case of the fine grained low permeability sandstone and of the chalk sample, permeability after acetate peel has significantly increased. This is due to the removal of flow restricting rock flour on the samples end faces. A large database exists which suggests that the litholigies most susceptible to end face rock flour damage are fine grained sands / silts and carbonates (matrix permeability).

Table 1. Comparison Of Air Permeability. Uncleaned, Brushed And Acetate Peel End Face Treatments.

	Permeability to Air (mD)		
Lithology	Uncleaned	Brushed	Acetate Peel
Fine Grained Sandstone	7.18	7.16	7.56
Coarse Grained Sandstone	297	296	303
Chalk	1.83	1.84	1.92

Continuous Immersion Soxhlet Cleaning

The main objectives of this new design are to enable efficient solvent cleaning of core samples without damaging delicate minerals such as fibrous clays. Evidence presented in this paper and in many previous publications have shown that delicate minerals can be damaged during drying[3,4,5,6]. Standard Soxhlet type cleaning involves many rapid drying cycles and where speed of cleaning is the most important factor the potential damage is unacceptable in most cases. Cold Static Solvent cleaning is probably the least damaging method but normally the time involved is prohibitive. A compromise is required which has all the non-damaging benefits of cold static cleaning and most of the efficiency of hot Soxhlet cleaning. The method developed is called Continuous Immersion Soxhlet (CIS) cleaning.

The CIS technique consists of a flask where solvent (any standard core cleaning solvents have been used) is boiled. The vapour rises and is directed in to a condenser which sits above the core sample chamber. Condensed solvent drips down in to the sample chamber but is directed to the base of the chamber and percolates up from the base. The chamber is heated and includes a stir bar which agitates the incoming solvent. Core samples are placed on a perforated shelf which sits a few inches off the bottom of the chamber.

An outlet at the top of the sample chamber, above the top of the highest core sample allows solvent to run off back in to the original solvent boiling chamber. The set-up allows for continuous circulation of solvent. In practice, fresh batches of clean solvent are introduced periodically. Throughout the process the core samples are in contact with warm clean liquid solvent thus enabling efficient cleaning with no damage to delicate minerals. Following the continuous immersion cleaning, samples can be dried by critical point drying or low temperature oven drying.

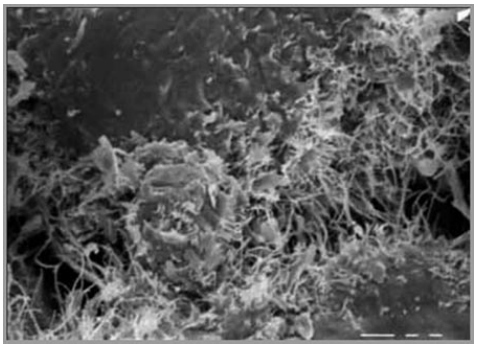

Figure 4 shows a sample cleaned using CIS and dried at low (60ºC) temperature. Delicate illite clay fibres are maintained in their original positions – no collapse has occurred. The sample and any subsequent measurements are representative of the reservoir.

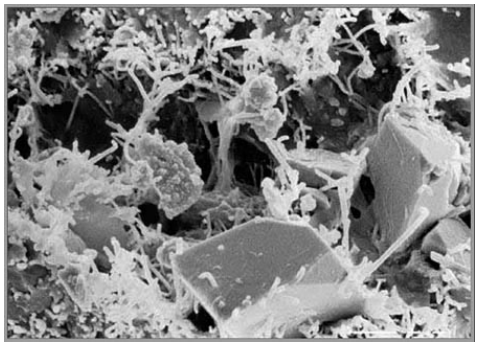

Figure 5 shows a sample cleaned using traditional hot Soxhlet cleaning and dried in a hot dry oven (120ºC). The illite clay fibres have collapsed and in some cases appeared "singed" from the high temperatures. This sample and any subsequent measurements are not representative of the reservoir.

Table 2 shows a selection of results from extensive testing of various cleaning and drying techniques. The data has been selected to demonstrate trends which have been observed but also to show anomalies which exist in most core measurement data sets. The data represents a comparison of standard hot Soxhlet cleaning and high temperature oven drying with CIS and lower temperature drying. All of the samples illustrated contain fibrous illite clay with percentages of bulk rock from 0.8% to 4.3% (determined by XRD with qualification by SEM). In general, the data shows that hot Soxhlet cleaning of fibrous illite clay rich samples leads to an increase in measured permeability. There is a reasonable correlation between the amount of this clay type and the observed increase in permeability. The main exception to this rules is sample 5 where the illite clay was later observed to have remained standing proud across all pores and no evidence of collapse was seen. Figures 4 and 5 are taken from sample 3 and clearly show collapse of illite clay as the reason for the increase in permeability. No changes in grain density were observed for these samples and small increases in porosity were sometimes observed.

Apparent porosity increases may be due to removal of tiny pores in between clay fibres due to collapse or might indicate migration of collapsed clay fragments.

Table 2. Comparison of Base Parameters - Samples cleaned and dried in conventional soxhlet / hot air drying and sample cleaned by C.I.S. and dried at 60°C.

Sample	Fibrous Illite as % Bulk Rock (XRD)	Porosity %		Grain Density g/cc		Permeability to Air (mD)	
		Conv	C.I.S.	Conv	C.I.S.	Conv	C.I.S.
1	2.1	22	22	2.64	2.64	317	294
2	0.8	25.6	25.5	2.65	2.65	380	382
3	2.9	18.4	18.1	2.65	2.65	60.3	48.4
4	4.3	21.2	21	2.67	2.67	13.9	10.2
5	1.8	16.5	16.4	2.65	2.65	19.4	19.4

CONCLUSIONS

Many techniques used in standard core preparation can affect the subsequent measurements made on those cores. This paper outlines two significant improvements in sample preparation which have been successfully developed and implemented. A selection of data and photographs presented in this paper have indicated that the use of these new techniques can have a major impact on petrophysical measurements. With continued vigilance and development we can help to improve understanding of reservoir properties and performance and produce laboratory data which accurately represents real reservoirs.

REFERENCES

1. Cornwall, C.: 2001. *The Preservation or Removal of Solid Bituminous Material, as part of the Core Analysis Programme on the Elgin-Franklin Field*: SCA 2001-48
2. Katz, A and Friedman, G.M.: *The preparation of stained acetate peels for the study of carbonate rocks*: 1965.
3. Pallatt, N. Wilson, J. and McHardy, B.: *The Relationship between Permeability and the Morphology of Diagenetic Illite in Reservoir Rocks*: 1984 SPE 12798.
4. Gant, P. and Anderson, W.: *Core Cleaning for Restoration of Native Wettability*: 1988 SPE 14875
5. Morrow, N. Cather, M., Buckley, J and Dandge, V.: *Effects of Drying on Absolute and Relative Permeabilities of Low-Permeability Gas Sands*: SPE 21880 1991
6. Mikkelsen, M. Scheie, A., Rong, Q and De Boer, E.T.: *Abnormal Permeability Behavior of a North Sea Sandstone Reservoir*: 1991 SPE 22600.