QUANTIFYING THE EFFECTS OF CORE CLEANING, CORE FLOODING AND FINES MIGRATION USING SENSITIVE MAGNETIC TECHNIQUES: IMPLICATIONS FOR PERMEABILITY DETERMINATION AND FORMATION DAMAGE

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ABSTRACT
Magnetic techniques have recently provided a rapid, non-destructive means of quantifying mineral content, particularly permeability controlling clays, at several core scales (Ivakhnenko and Potter, 2006, 2008; Potter, 2005, 2007; Potter and Ivakhnenko, 2008; Potter et al 2008, 2009). The correlation of magnetically derived clay content with key petrophysical parameters has lead to improved prediction of permeability and other SCAL properties. Magnetic techniques also offer a unique means of quantifying the effects of core cleaning. Some preliminary results at low field were reported by Potter et al (2004). The present study investigated the effects of hot soxhlet cleaning, by monitoring the low field magnetic susceptibility of the core before and after cleaning, on a series of well characterised samples. The work demonstrated that there was a reduction in paramagnetic illite clay content after cleaning, with intrinsically higher permeability samples exhibiting the largest reductions. Importantly, the results question the reliability of some permeability measurements made on cleaned (particularly intrinsically higher permeability) core samples, which undoubtedly overestimate the permeability of the uncleaned core with the original clay (and other mineral) content. Our techniques suggest a means of correcting for the effect of clay/other mineral removal during core cleaning. Magnetic measurements taken before and after cleaning could uniquely allow one to estimate the permeability of the uncleaned sample with the original mineral content.

We also detail sensitive magnetic techniques for quantifying fines migration and removal during core flooding experiments. A specially constructed coil has been used to measure the low field magnetic susceptibility at various points along the core both before and after core flooding experiments. Results showed that the low field magnetic susceptibility decreased after the experiments, implying that small concentrations of a mineral

(or minerals) with positive magnetic susceptibility had been washed out from the samples. Hysteresis measurements of the samples suggest (by analysing the low and high field parts of the hysteresis curves) that paramagnetic clay, and possibly some ferrimagnetic iron oxides, have been removed from the samples during these experiments.

On a larger scale, work on collected accumulated particulate residues from injection waters is further helping to build up a comprehensive picture of the nature and amounts of fines that might lead to formation damage. The particle size and concentration of iron minerals and clays removed are either generally too small for accurate XRD analysis or their signal is masked by high amounts of more abundant minerals such as halite or gypsum. The magnetic techniques, however, are very sensitive and can detect extremely small concentrations of the fines, which highlights a major advantage of this new approach.

INTRODUCTION

We have recently shown how magnetic susceptibility can be used as a useful additional tool for reservoir characterisation, quantifying mineralogy, and for predicting key petrophysical parameters in both clastic (Potter et al, 2004; Potter, 2005; Ivakhnenko, 2006; Ivakhnenko and Potter, 2006; Potter, 2007; Ivakhnenko and Potter, 2008; Potter and Ivakhnenko, 2008) and carbonate (AlGhamdi, 2006; Potter et al, 2008) reservoirs. The measurements provide a rapid, non-destructive complement to X-ray diffraction (XRD) for determining the clay content (Potter et al, 2004; Potter and Ivakhnenko, 2008), and for predicting permeability (Potter, 2005, 2007; Potter and Ivakhnenko, 2008) even in cases where the relationship between porosity and permeability is very poor (Potter, 2007). The measurements have also been shown to correlate with SCAL paramaters such as the cation exchange capacity per unit pore volume (Qv) and the flow zone indicator (FZI), as well as downhole wireline data such as the gamma ray signal (Potter, 2005, 2007).

The present paper will firstly detail how magnetic measurements can quantify the effects of hot soxhlet core cleaning. In particular we will focus on changes in illite content (an important permeability controlling clay) caused by the cleaning process, and the implications for obtaining accurate estimates of the permeability of reservoir samples. Schlachta (2008), using low field magnetic susceptibility measurements, has also reported the removal of quartz, kaolinite and ferrimagnetic particles from reservoir rocks by core cleaning. We will then describe how magnetic susceptibility measurements can be used to quantitatively monitor fines migration in core flooding experiments. This represents the first really practical way of quantifying such migration. Finally, we will show how collected accumulated fines in a larger scale water injection operation can be sensitively characterised by magnetic hysteresis measurements. These measurements have the advantage that they can detect extremely small amounts of paramagnetic and ferrimagnetic fines that are beyond the limit of detection of XRD. The hysteresis measurements can better quantify the fines that are passing through filters, and thus enable one to better assess the potential implications for formation damage in the reservoir.

QUANTIFYING THE EFFECTS OF CORE CLEANING USING MAGNETIC MEASUREMENTS

We have quantified the effect of hot soxhlet cleaning on a suite of sandstone reservoir core samples using low field magnetic susceptibility. We wanted in particular to see whether such cleaning removed permeability controlling clays. We undertook initial characterization of the samples using X-ray diffraction and chose samples that contained a mixture of quartz and illite clay with little or no other components. We further undertook initial magnetic hysteresis measurements using a Variable Field Translation Balance (VFTB, see Ivakhnenko and Potter, 2008) and chose samples that exhibited a straight line hysteresis graph (with no “kink” or “loop” at low fields) in order to ensure there were no ferrimagnetic mineral components in the samples. The low field magnetic susceptibility of the core plugs was first measured using a Molspin bridge, and the samples were then hot soxhlet cleaned using toluene and methanol. The low field magnetic susceptibility was measured again after the cleaning. The plug permeabilities were also measured after cleaning. The results are shown in Table 1. The advantage of the magnetic technique is that one can measure the same sample volume (plugs) before and after the cleaning, unlike XRD where one needs powdered samples and thus one can’t measure exactly the same sample volume. Also, since we chose samples with straight line hysteresis graphs, the high field magnetic susceptibility is exactly the same as the low field susceptibility (there is currently no simple high field bridge that allows one to use the same core plugs before and after cleaning as the VFTB ideally requires small powdered samples). Table 1 shows that the magnetic susceptibility is lower after cleaning for all samples. This means that a mineral or minerals with positive magnetic susceptibility has been removed from the sample. We can reasonably assume that this mineral is illite since our initial characterization and sample selection ensured that this was the main paramagnetic mineral in the core plugs. We calculated the illite content from the magnetic results before and after cleaning using the methodology of Potter et al (2004) and Potter (2007), thus quantifying the amount of illite removed due to core cleaning. These results are also shown in Table 1. The results depend on intrinsic permeability. Core plugs with a lower permeability show less of a reduction in illite content. The results have important implications for core plug permeability measurements, suggesting that cleaned plug permeabilities (particularly for intrinsically higher permeability samples) will overestimate the uncleaned plug permeabilities. The relation between magnetically derived illite content and permeability after cleaning closely followed that for another sandstone reservoir (Potter, 2007, Figure 2). Significantly, one could estimate the “uncleaned” plug permeabilities with the original clay content for the present samples by taking the illite content before cleaning and reading off the corresponding permeabilities from Potter (2007, Figure 2). Even better, the actual relationship between magnetic susceptibility (or magnetically derived clay content) and permeability after cleaning could be used (as each reservoir may exhibit a different relationship, especially if other clays are involved) along with the magnetic susceptibility (or clay content) before cleaning to estimate the permeability of the uncleaned samples. Using this approach the estimated permeability for an "uncleaned" Sample 1 in Table 1 would be 1021 mD (537 mD less than the "cleaned" permeability).

Table 1. Low field magnetic susceptibility and magnetically derived illite content before and after hot soxhlet cleaning of some N. Sea reservoir core plugs. Also shown is the permeability after cleaning.

Sandstone reservoir sample	**Mass magnetic susceptibility before cleaning (10^{-8} m^3 kg^{-1})**	**Illite content before cleaning (%)**	**Mass magnetic susceptibility after cleaning (10^{-8} m^3 kg^{-1})**	**Illite content after cleaning (%)**	**Illite removal from sample (%)**	**Permeability after cleaning (mD)**
1	-0.388	1.12	-0.412	0.95	0.17	1558
2	-0.381	1.17	-0.403	1.02	0.15	1124
3	-0.208	2.37	-0.216	2.31	0.06	117
4	-0.018	3.68	-0.026	3.63	0.05	73
5	0.442	6.86	0.438	6.84	0.02	11
6	0.586	7.86	0.584	7.85	0.01	1.8

QUANTIFICATION OF CORE FLOODING EXPERIMENTS AND FINES MIGRATION FROM MAGNETIC MEASUREMENTS

Magnetic measurements can quantify the effects of fluid flow and fines migration experiments very readily and sensitively. The magnetic techniques are possibly the only current means of studying and quantifying these effects at the core scale. XRD is not sensitive enough, and cannot measure the same (intact) sample of reservoir rock before and after the fluid flow experiment or procedure. Low field magnetic susceptibility sensors allow us to examine identical volumes of reservoir rock before and after the fluid flow. Low field measurements alone unfortunately do not allow us to differentiate between paramagnetic and any potential ferrimagnetic fines. However, these different types of fines can be distinguished and quantified very sensitively from hysteresis measurements on collected accumulated samples of the removed fines. A combination of the two techniques therefore provides a powerful new tool for quantifying fines migration and the effects of fluid flow experiments or procedures. We will describe work from laboratory core flooding experiments and a larger scale water injection operation.

Magnetic Characterisation and Quantification of Fines Migration in Laboratory Core Flooding Experiments

It is possible to measure the low field magnetic susceptibility at various points along a core plug using a specially constructed coil, the Bartington MS2C sensor (Figure 1 (a)). The diameter of the sensor closely matches the diameter of the core plug, and was built to our specifications by Bartington Instruments. The sensor allows measurements to be made at any given point along the core plug, and essentially measures the low field magnetic susceptibility of a thin slice (around 0.5 cm thickness) at each specified measurement point. A major advantage of this non-destructive technique is that measurements can be made of identical slices along the core plug before and after the core flooding experiments.

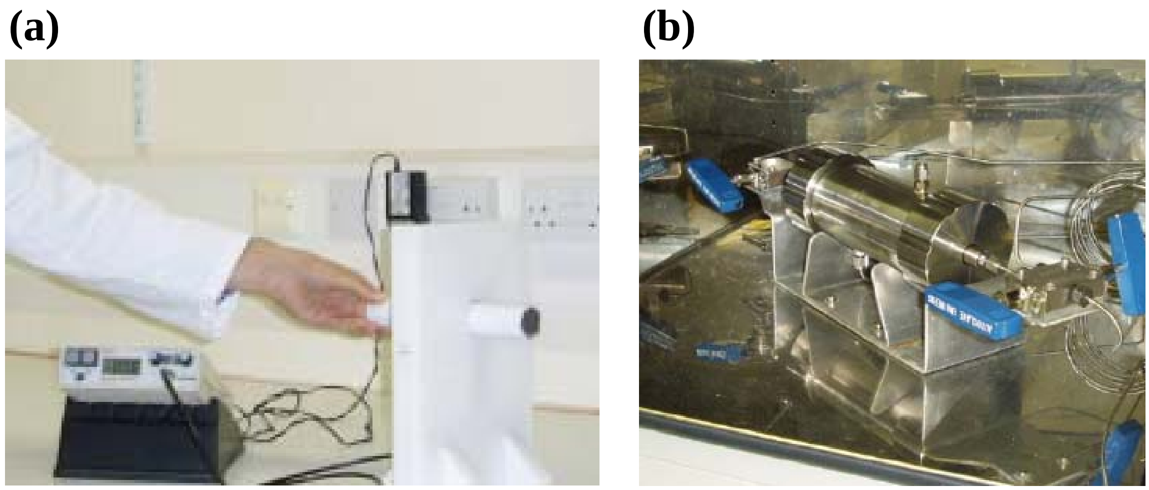

Figure 1. (a) Sensor (Bartington MS2C) for measuring low field magnetic susceptibility at points along a core plug before and after core flooding experiments. (b) The flow cell for the flooding experiments.

A flooding experiment using a seawater brine composition (around 24 g/l NaCl, 11.4 g/l $MgCl_2.6H_2O$, 2.3 g/l $CaCl_2.6H_2O$ and some other minor components) was undertaken on two reservoir sandstone core plugs of about 7.5 cm in length. The flow cell is shown in Figure 1 (b). The flow rate used in the experiments was 50ml/hour. The low field magnetic susceptibility was measured at various points along the cores using the MS2C sensor prior to the core flooding, and then again at exactly the same points after the core flooding. Figure 2 shows the results for both cores. At each measurement point the low field magnetic susceptibility decreased after core flooding (Figure 2 (a)). This means that a small quantity of a mineral (or minerals) with positive magnetic susceptibility was removed at each point due to the flooding. It is likely that illite clay was the main component that was removed during the flooding. We believe illite was primarily removed since this was the main clay mineral in these two core samples as indicated from XRD analysis, and was therefore the main source of fines with positive magnetic susceptibility. Note that whilst XRD is capable of identifying the clay composition in samples of the original rock, it doesn't have the advantage of the low field magnetic sensor technique in being able to measure the exact same piece of rock before and after the flooding experiment (since XRD needs to use a crushed powder sample). Also the small changes in mineral content before and after the flooding experiments are likely to be beyond the limit of detection of XRD. On the other hand one disadvantage of the low field magnetic susceptibility measurements is that they cannot indicate whether the fines removed during the core flooding comprise solely a paramagnetic mineral or a ferrimagnetic mineral (or contain both). Unfortunately there is currently no portable sensor coil (similar to the one in Figure 1 (a)) that measures a range of low and high field magnetic susceptibility values that would allow us the answer this question.

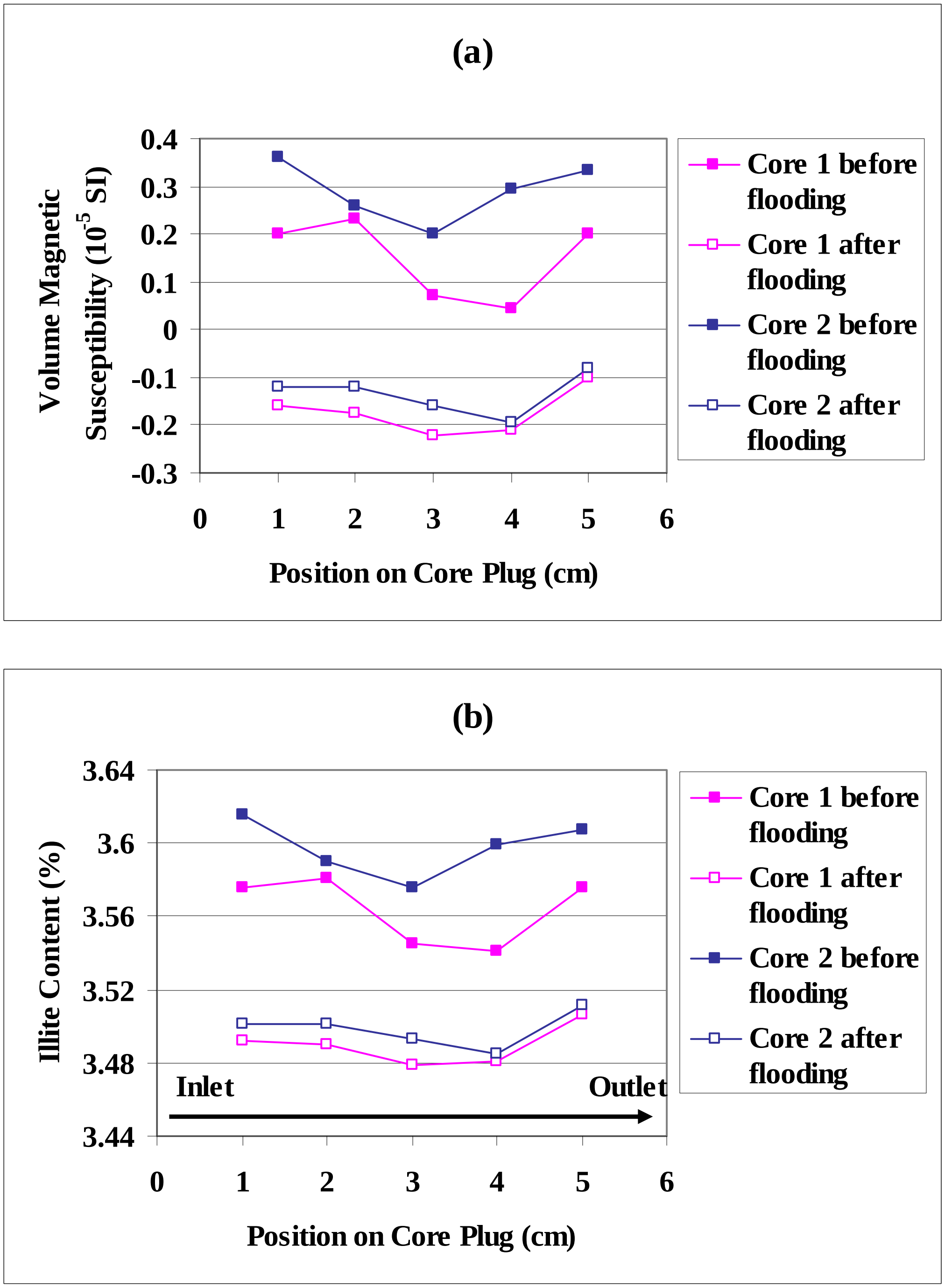

Figure 2. (a) Volume magnetic susceptibility determined at various positions (thin slices) on a sandstone core before and after flooding with a seawater brine compostion. (b) Corresponding plot of magnetically derived illite content asuming the core plugs comprised a simple two component mixture of quartz and illite. In each diagram the inlet was at position 0 cm and the outlet at position 7.5 cm. The throughput brine volume for Core 1 was 1140 ml and for Core 2 was 798 ml.

However, magnetic hysteresis measurements of small samples of these cores (Figure 3) indicated that they contained some paramagnetic clay (since the high field slope is higher than for pure quartz) with only extremely small amounts of ferrimagnetic material (as shown by the very small "kinks" in the curves at low field). This is further evidence for paramagnetic illite clay being the main component of fines that were removed due to the flooding experiments. This is even more likely in the case of Core 1 where the hysteresis curve is almost a straight line, indicating that it contains virtually no ferrimagnetic material. For Core 2, whilst it is most likely that illite fines are being removed, one cannot rule out the possibility that minute amounts of ferrimagnetic material are also being removed (or both).

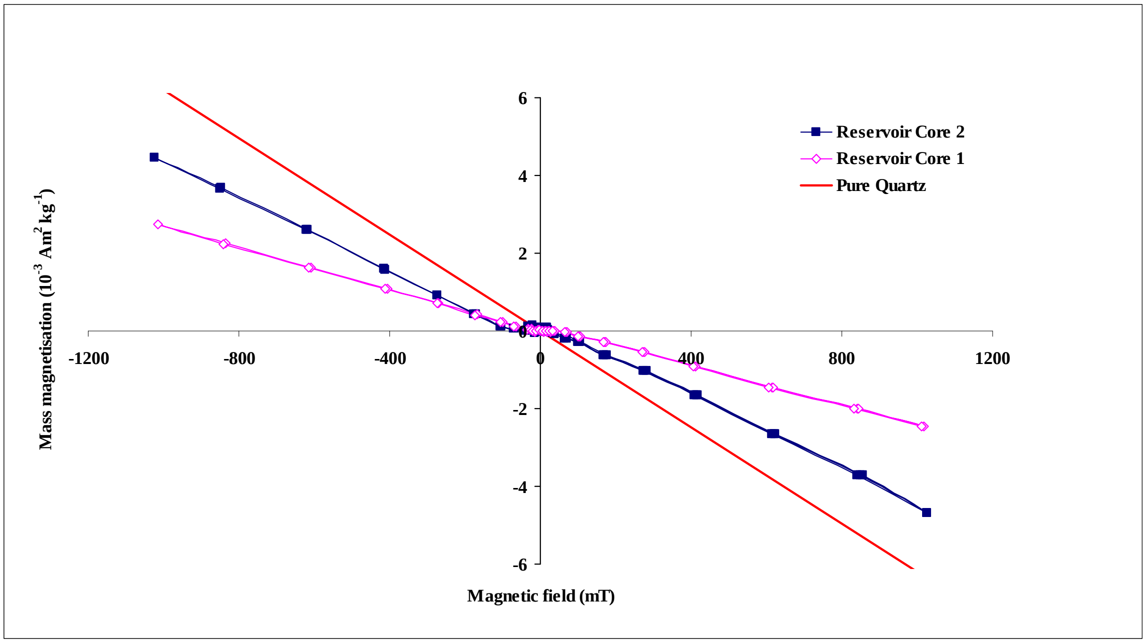

Figure 3. Magnetic hysteresis curves for small samples (at the outlet end) of the two sandstone reservoir cores used for the core flooding experiments. The curve for pure quartz is shown for comparison, and the horizontal (x-axis) would correspond to an illite content of close to 4% for samples comprising a mixture of quartz and illite. Core 1 is composed primarily of a diamagnetic mineral (quartz) with some paramagnetic material (XRD showed this to be illite) and virtually no ferrimagnetic material since the hysteresis graph is almost a straight line. Core 2 is again composed primarily of a diamagnetic mineral (quartz) with less paramagnetic (illite) content than Core 1 (as shown by the lower high field slope) but also contains an extremely small amount of ferrimagnetic material as shown by the slight "kink" in the hysteresis curve at low fields. Probe permeability at the outlet faces of Cores 1 and 2 gave values of 9.8 mD and 13.7 mD respectively.

To better characterize and quantify the removed fines we would ideally want to collect some of the fines and make magnetic hysteresis measurements on them. A possible way of doing this in future would be to collect accumulated fines on a filter paper placed at the outlet end of the core plug. A study in a larger scale water flooding operation, which tested the effectiveness of such measurements, is described below.

Magnetic Characterisation and Quantification of Collected Accumulated Fines in a Water Injection Operation

Magnetic hysteresis measurements were undertaken on the fines accumulated on filter papers placed at strategic points along a larger scale flow operation from a new injection line (water pipeline) in a North African sandstone reservoir. Filter cartridges are used to filter fines from the injected water in the pipeline in order to prevent formation damage in the reservoir rock. Figures 4 (a) and (b) show images of filter papers collected from the inlet and outlet of a filter cartridge at the manifold. The images show some slight staining due to fines, which is barely visible in much of the filter papers. The amounts of fines are below the limits of accurate detection by XRD. However, magnetic hysteresis measurements (Figure 5) using the VFTB are so sensitive that they were able to show clear differences between the filter papers. The virgin filter paper exhibited a straight line hysteresis graph demonstrating that it as diamagnetic with no ferrimagnetic impurities as expected. The filter paper that was located at the inlet of the filter cartridge exhibited the highest high field slope of the three filter papers tested, indicating that it contained the largest concentration of paramagnetic material. This concentration is still, however, very small. If the paramagnetic fines were illite then it would be equivalent to just 9 mg of illite in this case. The curve for the inlet filter paper also shows a distinct hysteresis loop at low field indicating a small concentration of ferrimagnetic material. This is not hematite, since the curve saturates at relatively low fields. Interestingly, the filter paper located at the outlet of the filter cartridge exhibits a high field slope that is between that of the virgin filter paper and that at the inlet (showing it has collected less paramagnetic fines than at the inlet), and a hysteresis loop at low fields which is smaller than that at the inlet (showing that the ferrimagnetic fines are less than at the inlet). This means the filter cartridge is reasonably efficient at removing fines, but clearly does not remove all the fines leaving the possibility of some formation damage to the reservoir.

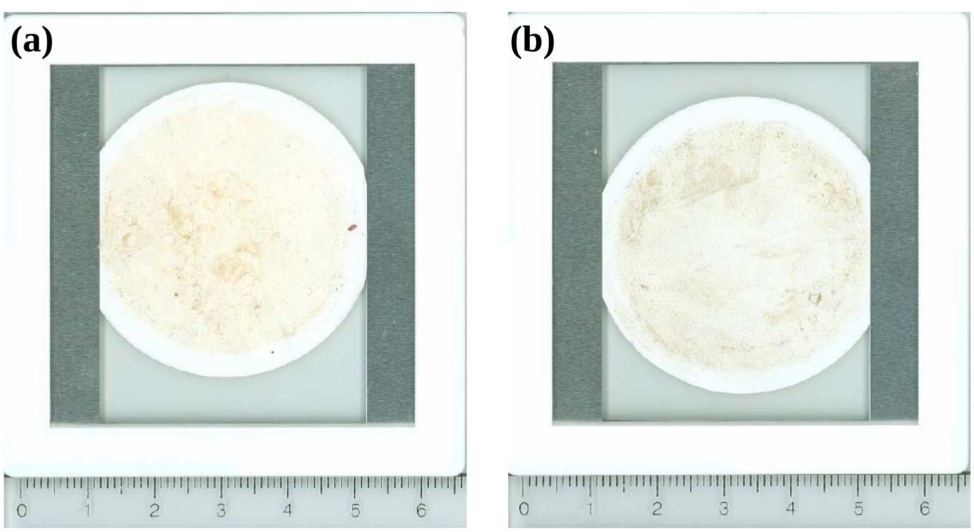

Figure 4. Image of fines collected on filter papers (a) at the inlet and (b) outlet of a filter cartridge at the manifold of a water injection pipeline at a North African clastic reservoir.

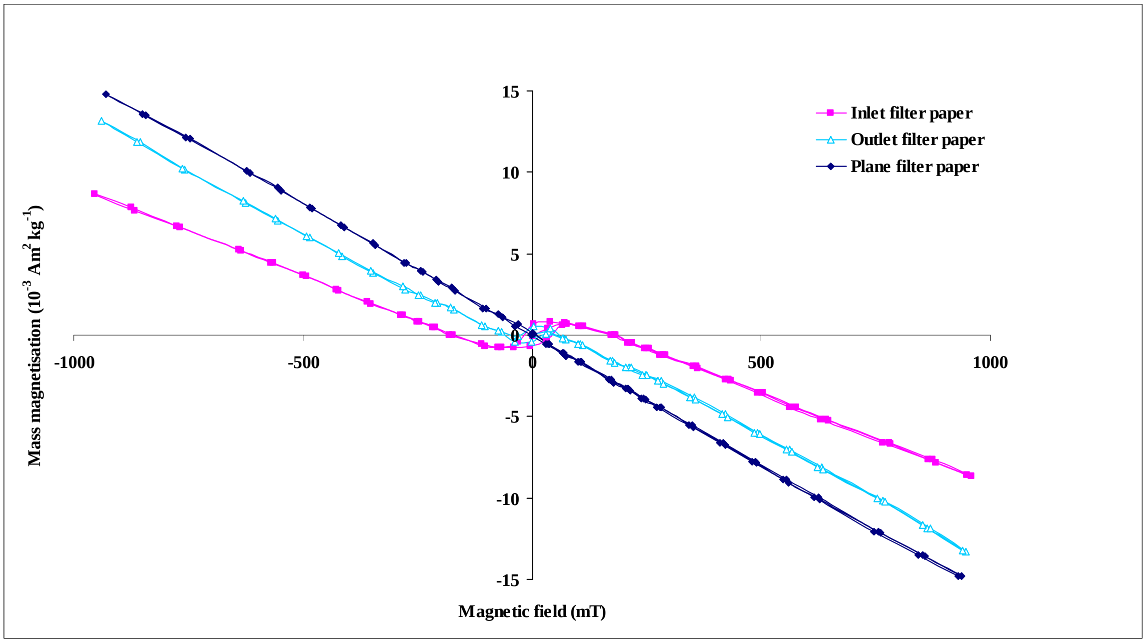

Figure 5. Magnetic hysteresis curves for samples of accumulated fines collected on filter papers. The filter paper located at the inlet on a filter cartridge at the manifold of a water injection pipeline has the highest high field slope (indicating the highest paramagnetic mineral content of the three filter papers), and also the largest low field hysteresis "loop" (indicating the largest ferrimagnetic content). The hysteresis results for the outlet filter paper (middle curve) indicate that it has a lower paramagnetic and ferrimagnetic mineral content. Also shown for comparison is the graph (essentially a straight line) for a plane (i.e., virgin) filter paper.

The above results suggest that filter papers could be used, on a slightly smaller scale, to trap fines at the outlet of core plugs in laboratory core flooding experiments. Magnetic hysteresis measurements of the filter papers plus accumulated fines potentially provides a very sensitive means of quantifying paramagnetic (clay) and / or ferrimagnetic (iron oxide) fines that may be washed out of the sample.

CONCLUSIONS

The main conclusions from this work can be summarised as follows:

- Magnetic susceptibility measurements before and after core cleaning uniquely allow one to quantify the effects of the cleaning process, since the same core plugs can be non-destructively measured before and after cleaning. In the samples studied the low field magnetic susceptibility was lower after hot soxhlet cleaning, which meant that a mineral or minerals with positive magnetic susceptibility had been washed out of the sample. In this case the relevant mineral was illite. The results have important implications for core plug permeability measurements, and strongly suggest that the permeability of hot soxhlet cleaned plugs is overestimated (particularly for intrinsically high permeability samples). The actual permeability of the uncleaned plugs with the original clay content would be somewhat lower.

- A means of estimating the original "uncleaned" sample permeability with the original clay content could be made by using the relation between magnetic susceptibility (or magnetically derived clay content) and permeability values after cleaning in conjunction with the magnetic susceptibility values (or magnetically derived clay content) before cleaning.
- Low field magnetic susceptibility measurements, using a special coil, can be used to quantify the effects of water flooding (and potentially other fluid flow) experiments at different points along a core plug. This can be done by making measurements at identical points along the core plug both before and after the flow experiment. The advantage of this is that the progress of fines at different points within the sample can be tracked, as there may be sections where fines accumulate and progress no further. Low field measurements alone are unable to distinguish whether paramagnetic (e.g., illite clay) or ferrimagnetic (e.g., iron oxides) fines or both are transported. However, hysteresis measurements, on a small sample of the core, can identify whether there are paramagnetic and / or ferrimagnetic components initially in the sample and thus indicate the potential types of fines that might be transported.
- In a larger scale water injection flow, magnetic hysteresis measurements were made on collected fines accumulated on filter papers at the inlet and outlet of a filter cartridge in a water pipeline. The measurements showed clear differences between the virgin filter paper and those at the inlet and outlet of the cartridge. They showed that some paramagnetic and ferrimagnetic fines were still obtained at the outlet of the filter cartridge, with the possibility that some formation damage could still be done to the reservoir sandstone. The amount and particle size of the fines were generally too small to be detected or quantified by XRD. This represents a major advantage of the magnetic techniques. The results also suggest that similar analysis could be applicable to the smaller scale laboratory core flooding experiments, as hysteresis measurements could potentially allow one to characterize and quantify paramagnetic and ferrimagnetic fines collected (on filter paper) at the core plug outlet.

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