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A NOVEL APPROACH TO TESTING CORE SAMPLES AT RESERVOIR CONDITIONS.

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ABSTRACT.

Diagenetic reactions associated with the injection of sea water for pressure maintenance and secondary oil recovery in North Sea reservoirs are poorly understood at present. This study describes the development and application of a unique core flood apparatus capable of reproducing *in situ* North Sea reservoir conditions in standard rock cores while flooding with fluids of different compositions. The basic design is a plug flow reactor which incorporates on-line high performance liquid chromatography (HPLC), allowing chemical analysis on small volumes of solution at the temperature and pressure of reaction. Results are presented which show how the permeability of core samples varies with the composition of the injected fluid. These show that the injection of sea water into reservoirs cannot be considered a chemically "passive" process, and in most cases, leads to a reduction in permeability.

INTRODUCTION

Laboratory core flood testing is a major component of oil field development, providing data that can be used to predict poroperm characteristics and evolution during production, assuming that appropriate parameters for scaling to the field are known. In order to minimise the physical variables during such scale up exercises, it is imperative that laboratory data on core plugs be obtained under conditions which reproduce those of the *in situ* reservoir environment, including temperature, stress state and fluid chemistry. In our bid to address this problem, we have built and commissioned a plug flow rig which allows a study of the evolution of petrophysical and chemical properties of reservoirs at core plug scale under the changing conditions of temperature, effective stress and pore-fluid chemistry. The need for such an apparatus arose from its potential application to the simulation of sea water injection for pressure maintenance and secondary oil recovery in North Sea reservoirs. The *in situ* temperatures in most North Sea reservoirs range from 80 to 130°C (Abbotts, 1991). However, most experimental studies on diagenetic reactions between sandstones and sea water have been conducted at much higher temperatures. For example, Hajash & Bloom (1991) reported some diagenetic reactions during semi-static flow-through experiments involving a feldspathic sand pack with sea water at 200°C. Both the confining and pore pressure were 100 MPa (14500 psi). Fluid chemistry and SEM studies of the solids showed that the reactions were dominated by the removal of Mg and Na from the fluid, resulting into the growth of smectite. An earlier study by Divis & McKenzie (1975), reported the growth of illite from K-feldspar during reaction of sea water with an artificial geosynclinal sediment at 200-300°C and 3 kbar pressure. In both studies, permeability dteremінations were not carried out. The thesis of these experiments is that sediment-sea water interaction is dominated by reactions leading to the growth of phyllosilicates at the expense of feldspars. In general, the chemical processes associated with the injection of sea water at low temperatures are poorly understood at present (Basan, 1985), partly because traditionally, water flooding has been considered a chemically "passive" operation whose role in formation damage is restricted to particle migration and dispersion (e.g. Jones, 1961). However, there is a growing awareness that the injection of any aqueous fluid into a reservoir that is at equilibrium with formation water of a different chemical composition initiates a range of chemical interactions that induce changes in the permeability of the reservoir (Bazin & Labrid, 1989). Unfortunately, the models used in such studies routinely eliminate aluminosilicate reactions, concentrating instead on reactions of clays and carbonate cements..

In this paper, we describe a re-circulating flow rig for simulating core flooding at reservoir conditions and present some results to demonstrate the relevance of such an approach. Emphasis on aluminosilicate reactions led to the choice of analogues containing mostly quartz and feldspar in our studies. The data so generated allows a correlation of the petrophysical changes within the core to the changes in the composition of the injected fluid.

DEVELOPMENT AND TESTING

Development

The design of a flow rig is dictated by the theories of flow operation which have been reviewed in detail by Posey-Dowty et al (1986). Usually, flow rigs used by geoscientists consist of a vessel/tube in which a reservoir core or sand pack acts as a packed/suspended bed and a fluid is passed through it during the course of reaction. Two types of flow characteristics are created dependent mainly on the magnitude of the axial dispersion. If the axial dispersion is large, the reactor is termed a continuously-fed stirred tank reactor (CSTR), the reverse situation yielding a plug flow reactor. Examples where packed bed reactors have been used include studies of Posey-Dowty et al (1986), Rimstidt & Dove (1986), Dove & Crerar (1990), Hajash & Bloom (1991) and Reed & Hajash (1992). The designs used in these studies, however, operate in semi-static mode, with large sample volumes being withdrawn and as Posey-Dowty et al (1986) point out, corrections for sample quenching, pressure drops and dilution must be applied to the data based on a number of assumptions.

Our flow rig has been designed on the basis of a plug flow reactor, but operates in a re-circulating mode in order to provide continuous mixing. The final design was dictated by the requirement that the rig should:

1. provide continuous re-circulating of a fluid through a standard rock core at *in situ* pressures and temperatures without any reverse flow or pressure pulses;
2. work with any fluid found in oil field environments and can withstand major corrosion by aggressive brines and organic acids;
3. allow analysis, on line, of the fluid chemistry on microlitre samples without resorting to quench techniques to avoid corrections to the data;
4. allow introduction of tracers in order to effect monitoring of fluid pathways and reaction sites in the rock using an ion microprobe;
5. be fully automated and hence provides continuous uninterrupted sampling.

In its final form, the rig consists of a biaxial core holder, a pair of fluid accumulators connected to the core holder and a pair of high precision pumps, all in a closed loop connected by 0.06" OD, 0.02" ID Polyetheretherketone (PEEK) tubing (Fig. 1). The reaction fluid is driven by a pair of stepper-motor M2-1000 Hydratron pumps supplied by Scotia Oil field Services Ltd. The pumping fluid is separated from the analytical fluid in isolation chambers machined in-house from high tensile stainless steel and fitted with PEEK liners and end plugs. One end of each chamber is connected to the pump and the other end to the core holder and its attendant valving. The top closure of the isolation chambers have been fitted with a 2 μm titanium frit to prevent plastic particles dislodged from the chamber walls from migrating into the flow lines. The pump and reaction fluids in the isolation chamber are separated by a PEEK slider fitted with an O-ring. During tests, one pump drives the fluid through the core into the second accumulator, while the other pump backs off at a rate that maintains both the volumetric flow rate and the pore pressure in the system to the set value. At the end of each stroke, the pumps reverse direction, with valve switching to maintain constant fluid flow direction. Because the core head pressure is normally higher than the tail pressure, the timing of the switch over must be such that no back-pressure pulses occur in the system. To achieve this bump-less transition, the tail pump raises the fluid pressure to match the pressure at the head side of the core. The fluid flow valve is then tripped and the pump directions reversed. The tail pressure is then reduced to the set value until the next cross-over is reached. Thus by measuring the pressure on the transducer on the input pump, the resulting differential pressure can be used to calculate relative changes in permeability.

The core holder was supplied by Edinburgh Petroleum Services (EPS) and came with a choice of 316 stainless steel or Hastalloy platens and nitrile rubber lining. The original platens were replaced by Grade 1 titanium platens machined in our workshop. To control fines migration into the fine bore tubing, the outlet platen of the core holder was re-designed to hold a filter stack. The original stainless end-piece supplied by the manufacturer consisted of a single bored stainless rod, with a stainless platen screwed to its inner end. The new design consists of a titanium platen with an extended rear threaded section, which screws into a chamber in a shortened backing rod. The filter stack is housed in this chamber, in the order (starting from the down flow side) 2 µm titanium frit, 0.45 µm polypropylene Millipore^{TM} or Hydrabond NH^+ filter, a stack of 5 µm nylon filters and another 2 µm titanium frit. Finally, a 316 stainless end shoe is screwed to the outer end of the titanium section to spread the end-load of the confining collar.

The fluid is analysed by using high performance liquid chromatography (HPLC) with a Waters^{TM} Action Analyser, employing conductivity detection for alkalis, alkaline earths and silica, and UV detection for transition metals. Sampling is automated by programming via a computer that manages the HPLC and all components attached to it, including detectors, a column heater controller, column heaters and air actuated rotary PEEK valves (Valves 2 and 3). The sample taken is typically 10-20 µl and can be reduced further. As the sample is diverted into the HPLC , the rig is automatically topped up by exactly the same volume of utrapure water from a wash line that is switched in and out of line through the valve sequences. Because of the small volume taken compared with the total volume in the isolation chambers (typically 200 ml) dilution correction is unnecessary. In addition, the analytical columns are maintained at the temperature of the reaction and the only time the sample experiences a slight drop in pressure is when it has already been mixed with the eluent. Correction for pressure and temperature drops is therefore also obviated.

The rig is capable of confining the core sample to 68.9 MPa (10,000 psi) and will also hold pore fluid pressures of up to 34.5 MPa (5000 psi), while being heated to a temperature of up to 100°C, constrained by the softening temperature (120°C) of PEEK tubing.

Flow Dynamics.

In order to confirm plug flow characteristics within the core holder, a pulse tracer test was conducted by injecting 100 µl of a 2000 ppm chloride via a sample loop in valve 2 and monitoring the concentration of chloride in the effluent exiting the core holder. The accumulators were by-passed and an HPLC pump was used during the test, allowing automatic and instantaneous injection of the tracer. Distilled water was pumped through the core holder containing a sandstone core with a pore volume of 12.61 cm^3 at 5000 psi confining pressure. The effective total volume in the core holder, including that in the filter housing was therefore 13.36 cm^3. At a pre-determined time ($t = 0$), the tracer was injected into the stream by appropriate switching of valve 2 and 1 ml samples of effluent were collected at intervals of approximately 4 minutes. The samples were diluted to 100 ml and analysed for chloride using HPLC.

In an ideal plug flow reactor operating in an isothermal environment with uniform composition and pressure distribution, all of the injected tracer should exit the cell at a time equal to the resident time of the reactor (Hill, 1977). However, the rig is operated in a re-circulating (recycle) mode, with a recycle ratio (volume of fluid returned/volume of fluid exiting permanently) of ∞, so that the rig behaves essentially as a mixed flow reactor (Levenspiel, 1979). In this mode, a step change in the concentration of an inert tracer should respond according to the material balance equation (Carberry, 1976):

$$\nu Co = \nu C + V\frac{dC}{dt} \tag{1}$$

where ν = volumetric flow rate in cm^3/min, Co = concentration of tracer in input solution in ppm, C = concentration of tracer in effluent in ppm, V = reactor volume (pore volume in core + filter housing) in cm^3 and t = evolution time in minutes. Integration of this equation gives

$$C(t) = Co\{1 - EXP(\frac{-\nu t}{V})\} \qquad (2),$$

subject to the boundary conditions that at $t = 0$, $C = 0$ and $t < 0$, $Co = 0$.

The residence time τ is the average amount of time a fluid element spends in the vessel and is defined as

$$\tau = \frac{V}{\nu} \qquad (3).$$

Substitution of equation 3 into 2 yields

$$C(t) = Co\{1 - EXP(\frac{-t}{\tau})\} \qquad (4).$$

For a tracer spike in a mixed flow reactor, the concentration-time curve is the differential of the step input function:

$$\frac{dC}{dt} = \frac{CoEXP(\frac{-t}{\tau})}{\tau} \tag{5}$$

The results of our tracer spike test (Fig. 2a) are compared with theoretical time-dependent changes in effluent tracer concentration generated by equations 4 (Fig. 2b) and 5 (Fig. 2c). Experimental results show a spread of residence times with maximum chloride in the effluent at 44.5 minutes, the average residence time for the effective volume in the core holder. This confirms plug flow behaviour and the spread in residence time is a natural consequence of back-mixing of fluid elements as a result of axial dispersion and turbulence/eddy diffusion arising from the influence of pore geometry in the core sample.

According to equation 4, a steady state composition of chloride in the effluent, equal to 2000 ppm should be reached in exactly 6 hours of flooding in a well mixed reactor. In one of our tests (Test #8), where brine injection followed soaking in distilled water, the sulphate concentration in the fluid is shown to reach steady state somewhere between 6 and 25 hours (Fig. 4b) , with a time-dependent trend similar to that generated by equation 4.

EXPERIMENTS

Sample Preparation and Characterisation

So far, we have used cores of the Clashach sandstone, an onshore analogue of some North Sea reservoirs. Cores measuring 2½-3 inches long and 1½ inches in diameter were taken from sandstone slabs. At the same time, samples were taken from the same slabs for characterisation which included petrographic studies, scanning electron microscopy (SEM), electron microprobe analysis and X-ray diffraction (XRD). In addition, porosity and permeability was measured for the core samples, although this was not done for Test #1 and Test #6. These studies show that the Clashach sandstone is a pale fawn, well sorted, medium to coarse grained (0.5-1 mm) subarkosic arenite. Quartz ranges from 90 to 96% and K-feldspar makes up the rest, with trace amounts of illite. Occasional sections show traces of ferroan dolomite and kaolinite. Todd et al (1990) also reported chlorite, although this has not been detected in our samples. The permeability measured for different samples was variable, ranging from 200 mD to 1D. The low permeability samples, in general show a higher degree of red colouring, suggesting that the observed variation is due to pore-throat blockage by ferric compounds.

Prior to loading in the cell, the cores were dried in an oven and weighed. In order to remove air in the pores, the cores were soaked overnight, often in the flooding fluid, except for Test #8 where the core was soaked in distilled water. They were then cleaned of surficial fines by utranonicating them for 5 to 10 minutes, loaded in the core holder and the confining pressure raised to 5,000 psi. The pore pressure was then raised to 3,000 psi, except for Test #1 where the sample was flushed with distilled water before the pore pressure was applied.

Flow Experiments

Results presented here are for experiments in which the core was confined to 34.5 MPa (5000 psi) and the pore pressure was maintained at 20.7 MPa (3000 psi), giving an effective stress of 13.8 MPa (2000 psi). The rig was then heated to 80°C while flooding with fluids of different compositions over durations ranging from 2 to 5 weeks. In this case, the 80°C limit was imposed by the necessity to conduct chemical analysis at the temperature of reaction using columns rated to this temperature. From a survey of literature and discussions with operators, a volumetric flow rate of 0.3 cm^3min^{-1} was chosen for all tests. The fluid was sampled at regular intervals and analysed for by on-line HPLC. Occasionally, off-line samples were taken in a nitric acid eluent for cation analysis by atomic absorption spectrometry, particularly where high analyte levels would overload HPLC columns and produce poor chromatographic data.

Although the emphasis is to study the effects of injecting sea water in reservoir sandstones we found that the HPLC columns could not cope with the high levels of Mg^{2+}, Na^+, Cl^- and SO_4^{2-} found in sea water, even if a small enough sample (2 µl) was taken. In order to meet the criteria for on-line analysis, we used artificial brine solutions made according to the recipe of Harrison et al (1980), but in which the Mg^{2+}, Na^+, Cl^- and SO_4^{2-} concentrations were reduced. This is justified for Mg^{2+} because Hajash and Bloom (1991) reported that reducing the Mg^{2+} content of their starting fluid had no effect on the nature of reactions that occurred in their experiments. We have no previous information, however, on the other three species as to how reducing them would affect overall behaviour of the fluid-rock interactions. In addition, we include in our discussion results of pure water flooding, mainly because they provide a reference framework for brine tests, but also because they reveal some valuable insights into the relative roles of particle migration and chemical processes on permeability changes.

RESULTS AND DISCUSSION

Injection with Brine Solutions

Two tests, each of five weeks duration, were conducted on the Clashach sandstone using artificial brine solutions with an initial pH of 7.2. In Test #7, the fluid evolved by losing about 70 ppm Mg^{2+}, the loss starting after 1 week of reaction (Fig. 3a). At the same time, Ca^{2+} increased in the fluid from 390 to a maximum of 460 ppm (Fig. 3b). Further flooding led to uptake of Ca^{2+} by the rock to more or less the original brine composition. There was virtually no change in the K^+ content of the fluid in the first 400 hours, but the fluid lost about 50 ppm after that (Fig. 3a). No significant change in the sulphate content was observed, other than a slight increase between 350 and 500 hours (Fig. 3b). The on-line permeability dropped to 87% of the initial value in the first 24 hours (Fig. 3c). This was followed by a more gradual decline throughout the duration of the experiment, although the permeability apparently stabilised at about 70% of the initial value in the second half of the experiment. A minipermeameter traverse for this sample was conducted after the core had been flooded. The traverse shows low values at the inlet end and in the middle of the core and high permeability intervals between 15 and 35 mm and between 45 and 55 mm (Fig. 3d).

Except for the initial increases during the first 100 hours, the composition of the brine in Test #8 stayed more or less constant for 600 hours. This stable compositional stage was followed by sudden loss of Ca^{2+} and SO_4^{2-} from the fluid from about 600 hours to the end of the experiment, and Mg also declined slightly (Fig. 4a and b). The on-line permeability dropped to 75% of the initial value and then gradually decreased to about 45%, with little evidence of stabilisation towards the end (Fig. 4c). Minipermeameter traverses were conducted both before and after flooding (Fig. 4d). The pre-flood traverse starts at 240 mD at 5 mm and rapidly rises to 480 mD at 10 mm, before dropping gradually towards the outlet end of the core. The post-flood traverse depicts a similar trend to the pre-flood traverse, but all points in the post-flood traverse plot below those of the pre-flood, confirming the permeability drop recorded by differential transducers.

Injection with Distilled Water

Two experiments were conducted in which cores were flooded with distilled in order to investigate the effects of changing the fluid composition on both fluid chemistry and permeability. In both experiments, the initial fluid pH was 5.7, being equivalent to that of pure water equilibrated with atmospheric CO_2. However, in order to assess the relative contribution of particle migration and mineral growth/dissolution to permeability changes, the two experiments differed slightly in their set up. In Test #1, the sample was flushed overnight with distilled water before it was pressurised to test pressure and the experiment was carried out at room temperature (20°C) for a period of 2 weeks.

For Test #1, the calculated permeability has doubled (Fig. 5a) over the two week duration (100% increase). Elphick et al (1992) attributed this increase to dissolution of K-feldspar overgrowths in the sample. Although small, the changes in the Mg^{2+} and Ca^{2+} concentration of the fluid are rapid, achieving steady-state within 150 hours of flooding (Fig. 5b). For Test #6, the permeability of the core initially dropped to 12% of the original value (Fig.5c). It then rose steadily to 62% of the original value at about 350 hours before dropping back to 40% at 430 hours. A further rise to 63% at the end of the experiment followed this drop. The similarity in the trend observed for this sample to those of Sayegh et al (1990) is striking, and we interpret the initial permeability drop to be due to clay swelling and migration as this sample was not flushed prior to flooding. This interpretation is based on the fact that the initial drop in permeability is opposed to changes in solution composition (Fig. 5d) which suggest feldspar dissolution, as shown by the increase in the K^+ and SiO_2 content of the fluid.

The major difference between brine and distilled water experiments is the increase in permeability observed when cores are flooded with distilled water. In the past, experiments using distilled water have shown a reduction in permeability of core samples (e.g. Aruna, 1976). The general interpretation of such observations has been to attribute the decline to pore throat blockage by migrating particles and dispersed clays (e.g. Jones, 1961). However, Potter et al (1981) have argued that some of the reported permeability reductions can be attributed to fouling by ferric hydroxides generated by corrosion of stainless steel reaction vessels. In both our experiments using distilled water, we have recorded increases in permeability of cores. These apparent descrepancies may be due to our use of an ultra-inert core flood system where corrosion of lines in contact with fluid is minimised. The differences in the permeability trends can be attributed to sample preparation procedures. Thus when the sample was flushed before being pressurised (Test #1), we recorded a continuous increase in permeability. On the other hand, the core in Test #6 was not flushed prior to pressurisation and suffered an 88% reduction in permeability in the first 40 hours, recovering only to 63% of the original value. In tests where cores were flooded with brine solutions (Test #7 and Test #8), cores initially suffered respective drops in permeability of 13% and 25%, despite the fact that none of the cores were pre-flushed. Clearly, clay dispersion plays an important role in the overall permeability characteristics in distilled water. As permeability damage due to particle invasion and migration stabilises with time (Todd et al 1990), the rest of the permeability damage (17% in Test #7 and 30% in Test #8) to the end of the flood tests occurs gradually and can be attributed to chemical reactions occurring within the core. Thus sea water injection cannot be considered a chemically passive process, even in non-carbonated sandstones. Experiments such as those of Bazin and Labrid (1989) which eliminate aluminosilicate reactions need to be modified to take into account these important processes.

CONCLUSIONS.

An ultra inert flow rig has been constructed for core flood tests with the capability to provide on-line analysis of small sample volumes at reservoir conditions, obviating the need to correct the data for dilution, pressure and temperature associated with most sampling strategies available. Application of the rig has shown that the permeability changes observed during core flooding are strongly dependent on the composition of the injected fluid. In particular, injection of brine solutions has so far produced declining permeability with time, while distilled water injection yields the reverse. In both cases, the observed changes in permeability can be correlated with processes of mineral dissolution or precipitation as revealed by time dependent changes in the composition of the re-circulating fluid, in addition to changes induced by pore throat blockage due to particle migration and deposition. An important aspect shown by our experiments is the finding that sample preparation can produce large differences in the overall permeability characteristics of core samples. This calls for the need to standardise treatment procedures, without which all core flood data must be treated with extreme caution. Finally, it is important to emphasise that sea water injection cannot be considered a chemically passive process. As such, more experiments are needed in order to further constrain the chemical reactions associated with such an important operation.

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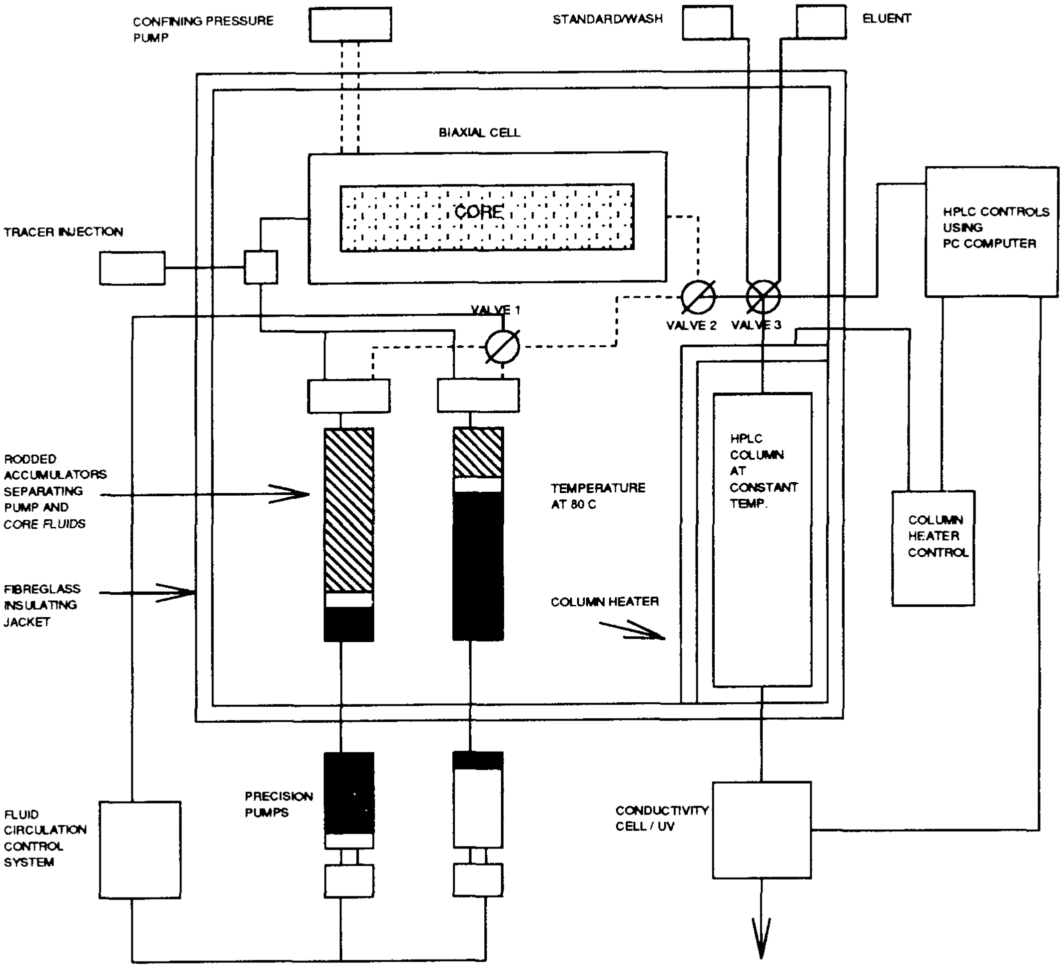

Figure 1. Schematic layout of the re-circulating flow rig. Valve 1 is the fluid flow valve that ensures unidirectional flow through the sample while valves 2 and 3 are for sampling. Sampling is automated from the HPLC.

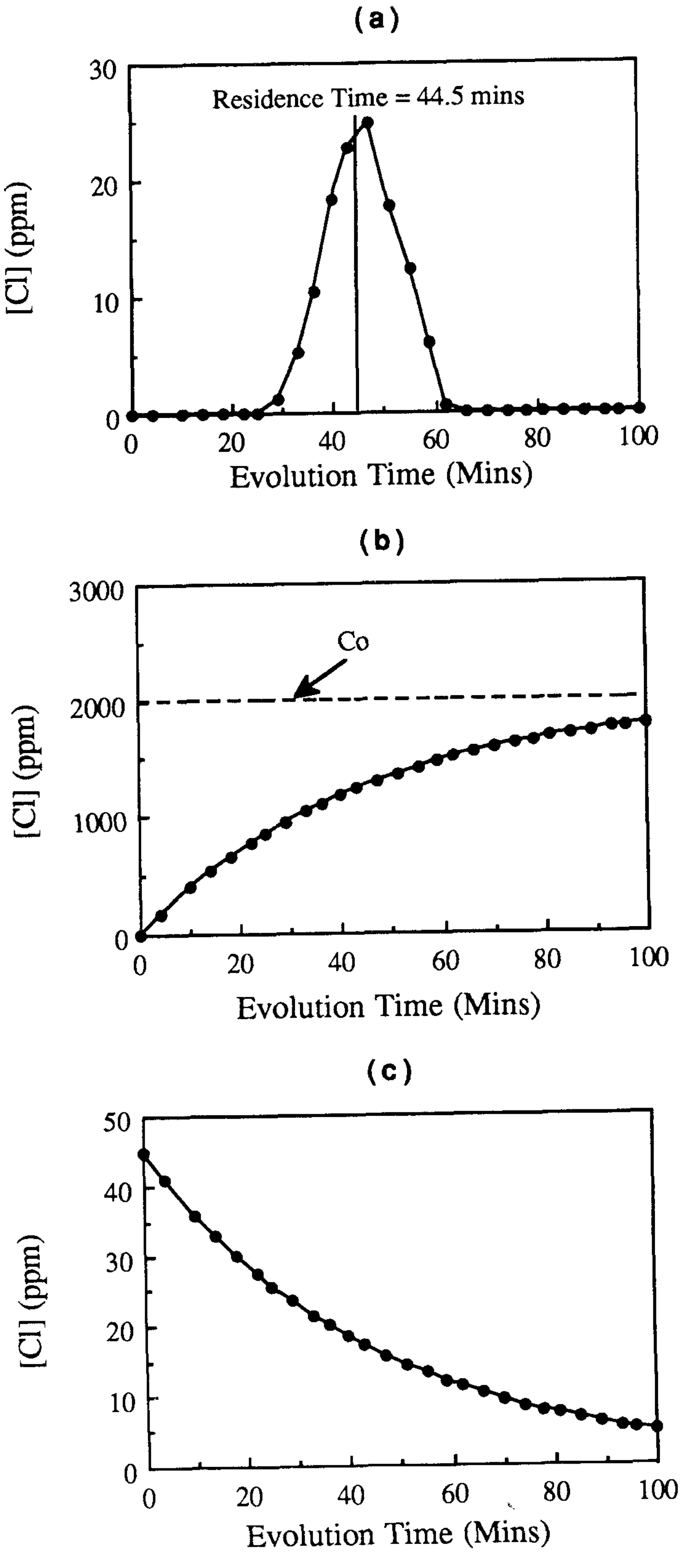

Figure 2. Chloride profile (2a) during pulse tracer testing. Figure 2b is a theoretical profile expected for a step input function in a well-mixed flow reactor and 2c is a corresponding theoretical profile for a pulsed injection in a well-mixed flow reactor. The experimental tracer test corresponds to plug flow within the core holder, with some dispersion caused by fluid mixing as a result of pore geometry within the core sample.

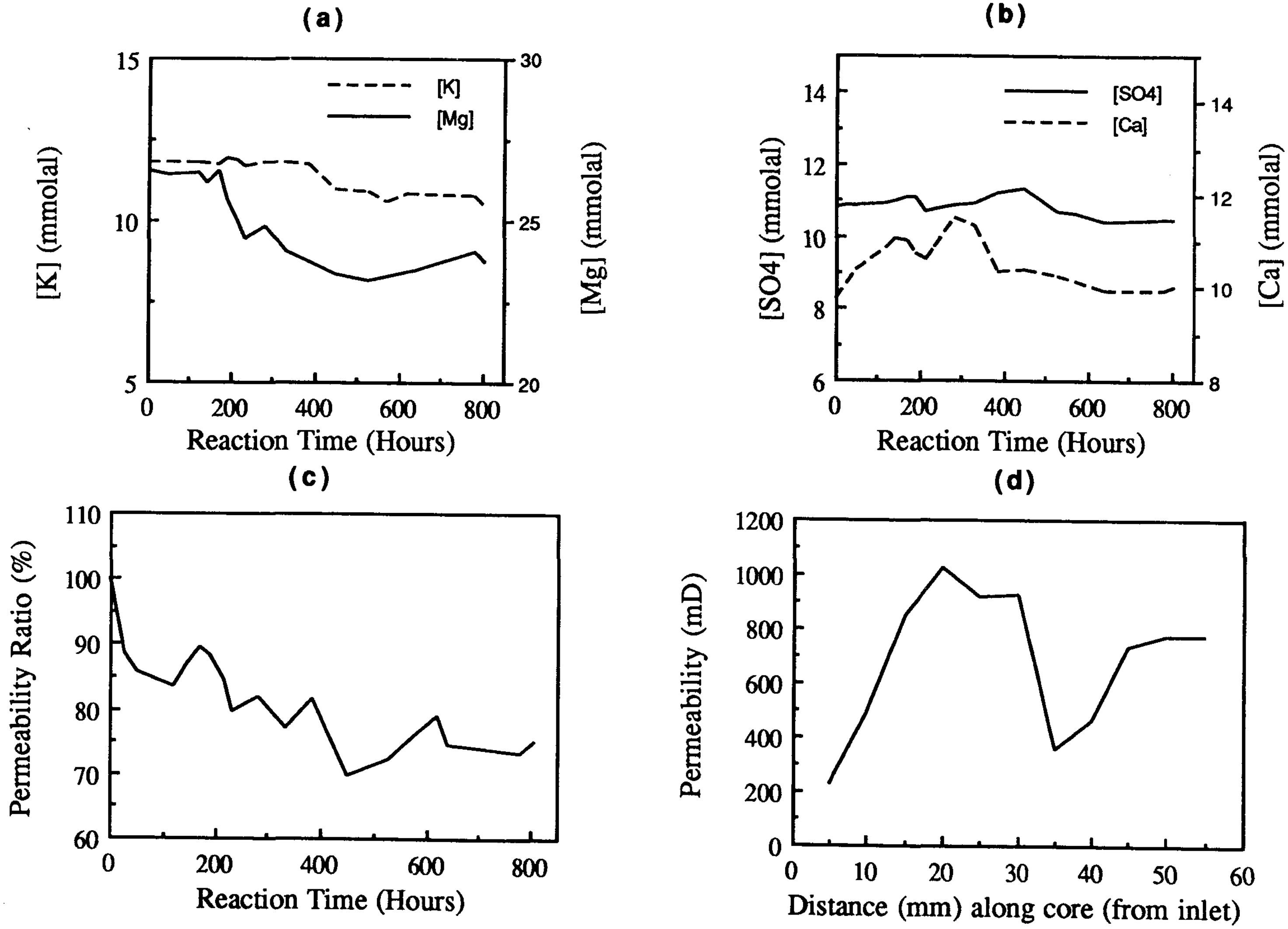

Figure 3. Time-dependent changes in the composition of the fluid (a and b) and permeability (c) during brine injection in Test #7. Note the steep decline in permeability during the early hours of flooding, which is due to particle migration and deposition. The minipermeameter traverse (d) shows some natural variation in permeability along the core.

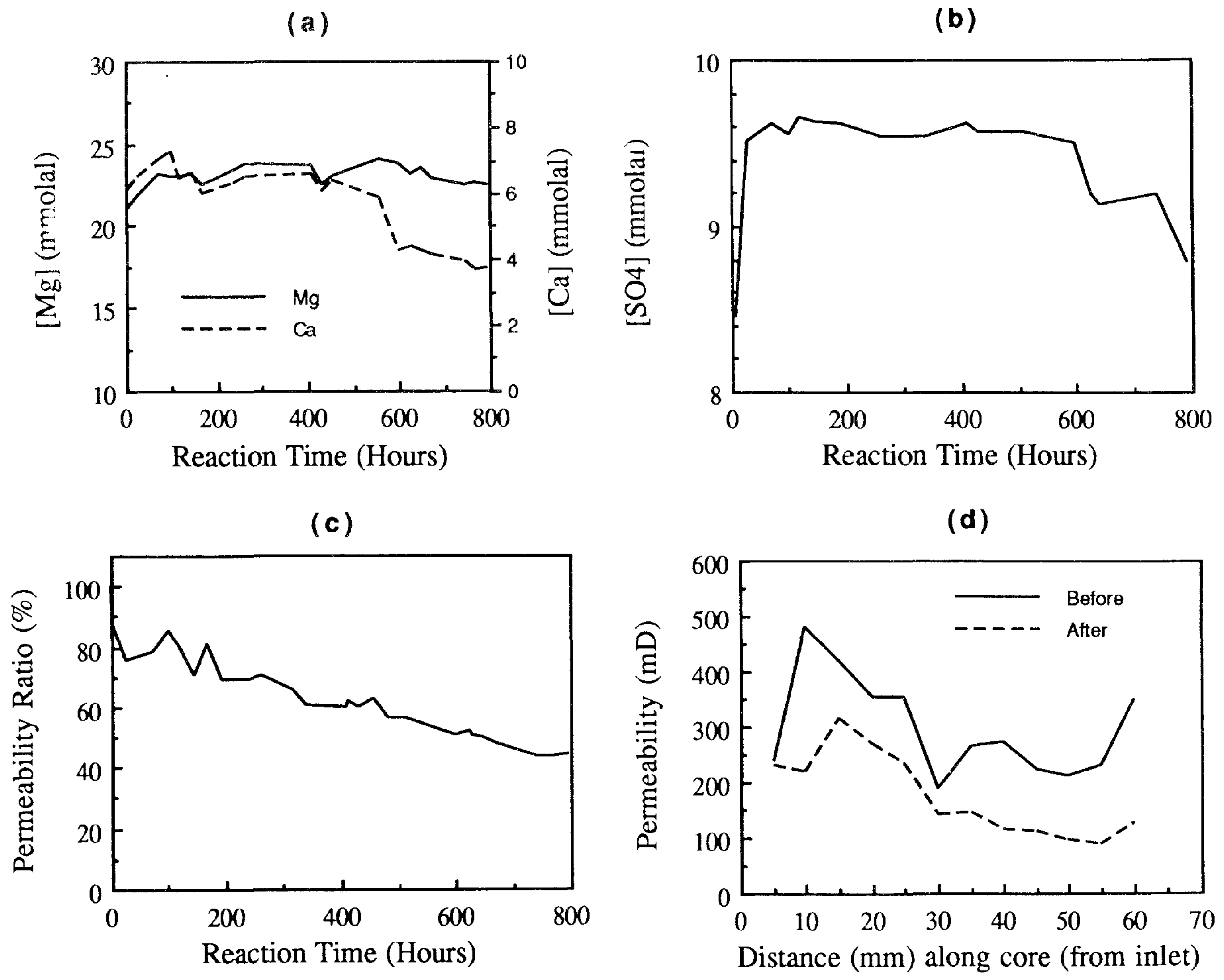

Figure 4. Changes in solution composition (a and b) and permeability (c) during brine injection in Test #8. The permeability trend is similar to that in Test #7. Minipermeater traverses (d) show that after flooding, the permeability of the core has decreased throughout the length of the core.

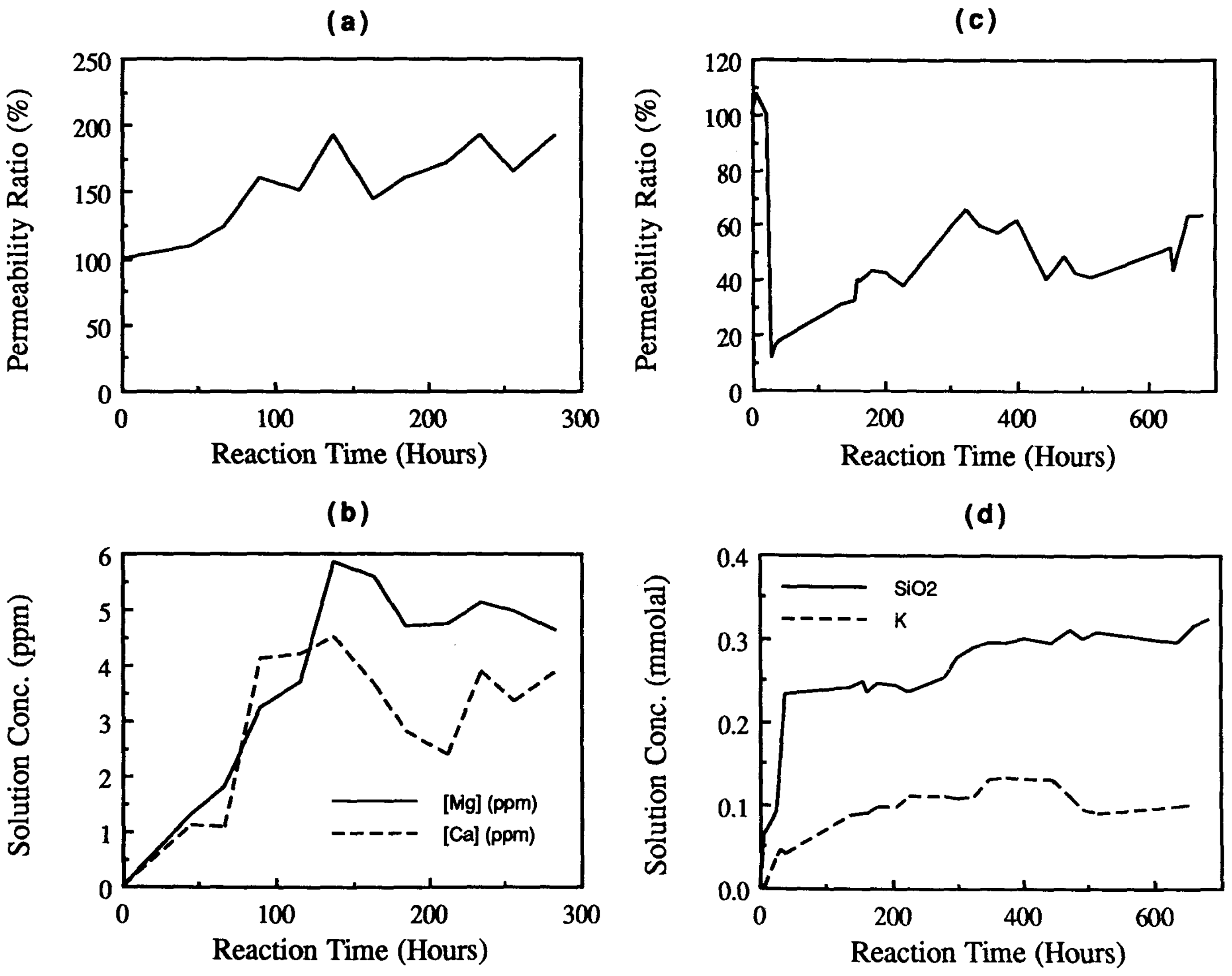

Figure 5. Permeability changes and solution composition during distilled water injection. In Test #1 (a and b), permeability increased continuously due to dissolution of trace dolomite and feldspar. Note also the sharp initial drop in permeability in Test #6 (c) where the sample was not flushed prior to pressuring. The rest of the test is dominated by an increase in permeability as feldspar dissolves (d).