# HPLC Chromatography Packing as a Tool to Evaluate Mechanisms of Formation Damage by Fines Migration

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### **ABSTRACT**

The aim of this paper is to show that a system of steel packed columns of known blends of minerals (with controlled ratios and size distributions) coupled to an equipment of high performance liquid chromatography (HPLC) allows us to emulate fines migration phenomena in rock porous media.

Time / pressure measurements were taken in two different ways:

☐ Fixed rate of injection with decreasing salinity to fresh water.

☐ Fixed salinity with variable rate of injection.

The results obtained by this non conventional dynamic method are similar to those in the case of displacement test in core plugs. Moreover, time of analysis is shorter and the technique is more simple.

## INTRODUCTION

Fines mobilization in oil reservoir represents a real headache for oil industry because it leads to drastic and undiscrable permeability changes during oil production<sup>1</sup>. In order to understand the release mechanisms of fines from pore network, is important the support of data provided by a good experimental tool.

Current laboratory experiments deal with displacement tests in core plugs<sup>2</sup> and also with visualization of this phenomena in micromodels<sup>3</sup>, where parameters like pore geometry and size, mineralogical composition of the grains and chemical composition of the present fluids are in most cases not controlled parameters. These problems can be overcome with sandpacks inside a chromatographic column.

Another important factor is the time consumed in the experiments, generally in terms of hours. The necessity to simplify and reduce time experiences, keeping reliable data, led us to implement the use of high performance liquid chromatography (HPLC) instruments with steel packed columns of known blends of minerals with controlled ratios and size distributions which emulate the rock porous media.

Gas and liquid chromatography<sup>4,5</sup> techniques are based in the fact that a porous medium, fixed inside a column, separates the components of complex mixtures that pass through it together with a carrier fluid.

This separation is based on surface interactions phenomena. The heart of a chromatograph (Figure 1) is the column and it is usually tested in two different approaches. One is related with separation efficiency values like the number of theoretical plates, resolution and peak symmetry. The other approach deals with its physical properties such as permeability, porosity and size of packing particles. For a core analyst, the later properties are of interest, even more when the composition of the packing is silica.

First of all, it is necessary to establish basic differences and analogies between a chromatographic column and a core. Uniform and small size particles and high pressure packing allow to get uniform and constant permeability and porosity, which leads to a good separation in chromatography. Is typical to have a nonuniform size particle as well as nonuniform porosity and permeability in a core. These inhomogenieties result in damage when noncontrolled external fluids pass through it. Monitoring of any changes in the permeability of the sandpack, in the column, with a liquid chromatograph is very simple due to the facilities the equipment has. It is for this reason that a steel chromatographic column has been packed with Ottawa sand and a small percentage of kaolinite and then put into a HPLC instrument to accomplish three specific objectives:

 To develope a reliable experimental scheme (equipment and procedures) to study fines related permeability decline.

- 2. To establish the existence of both hydrodynamical and chemical damage mechanism.
- 3. To provide guidelines of each damage mechanism.

Unconsolidated sandpacks were considered in earlier papers of Gruesbeck and Collins<sup>6</sup> and Gabriel and Inamdar<sup>7</sup>. Nevertheless, these authors did not use this type of sandpacks coupled to HPLC equipments.

In many respects, chromatographic columns can be likened to sandpacks. Both are composed of SiO<sub>2</sub> based materials and obey the same physical principles (Darcy's law) in their behaviour.

# **EQUIPMENT AND MATERIALS**

Flow tests were performed at room temperature, using unconsolidated sandpacks (600 — 900  $\mu$ m) with a small percentage of kaolinite (aprox. 30  $\mu$ m). Packing pressures ranged from 3000 to 5000 psig. The blend initially saturated with 3% of brine was introduced by means of an air driven pump, inside 316 steel chromatographic columns (0.7 cm i.d. 9.2 cm length), (Figure 2).

Data were obtained from a conventional HPLC instrument with programable rate and delivery pumping system where the columns were placed. Time (minutes) and pressure (atm) measurements were taken in a sequence of experiments controlling alternatively velocity and brine eluent concentrations (Tables 1,2). Fluids consisted of sodium chloride, barium chloride and distilled water at room temperature. SEM and a laser particle counter were used to measure particle sizes involved.

#### RESULTS AND DISCUSSION

Figure 3 shows the evolution of permeability as a function of the different volumes of brine injected at fixed flow rate. It can be observed that BaCl<sub>2</sub> brine behaviour is very similar to the one obtained in core plugs displacement tests<sup>8</sup>, this no decline in permeability could be explained by strong particle attachment on sand grains surface induced by divalent cations<sup>8</sup>. In the case of NaCl brine, the permeability evolution curve obtained is also similar to the one obtained in the conventional displacement test (Figures 3 and 4). It can be observed that with our methodology the value of critical

salinity concentration, CSC, is around 0.3% NaCl (conventional displacement test in berea sandstone in our laboratories yield a 0.27% NaCl as CSC value) and within the experimental error, it is an acceptable result.

The fundamental difference between our method and the conventional one is related with a decline of the permeability of the samples after the injection of certain concentration of brine (aprox. 1.5% NaCl), which produces a permeability loss of about 10%. The possible reason of this difference is the dynamic approach of our experiments, that would not allow the brine concentration to be equal along the column at the same period of time. In the conventional displacement test, the amount of brine injected (several pore volumes) is enough to ensure the same brine concentration along the core length.

It is important to remark that once the columns are placed in the chromatograph, the pressure recorded is very high (270 atm). For this reason the fluid flow is inverted and a steady pressure of 24 atm is maintained. This pressure allows us to calculate the initial permeability which belongs to the kaolinite piled up upon the 0.2  $\mu$ m column frit, as a result of the high packing pressure procedure.

The column used for NaCl salinity variations studies was also used to study the influence of flow rate variations on the sandpack permeability at fixed salinity, under dynamic conditions (Table 2). Figure 5 shows the evolution of permeability as function of brine flow rate. It can be observed that initially an increase in the flow rate of brine produces an increment in the permeability until a maximum is reached. A subsequent increasing of flow rate results in a decrease of permeability.

The initial increase of permeability occurs because in this case, one has a suspension of small fine particles flowing through a porous medium having large pores and there is plugging deposits would not be expect, as stated by Gruesbeck<sup>6</sup>, the type of entrainment of fines is the surface type deposit. The final decline on permeability is due to the accumulation of fines on the restriction imposed by the column frit as it was observed after the opening of sandpack when the test was performed. Maximun velocity of fines migration inside the column depends on the present kind of restriction. Large pores will permit higher velocities regardless of salinity conditions. This decline on permeability seems to

indicate that fines in a sandstone formation migrate distances longer than expected. Phenomenological description of these results is provided in Figure 6.

Finally, it is important to indicate that whereas this type of analysis takes 40 minutes, the conventional displacement test lasts about 8 hours.

#### CONCLUSIONS

It is possible to reproduce the phenomena of fines migration inside a packed colum acting as a closed system for particles and not for fluids. These columns work as a reversible system and indicate that fines can travel long distances, pore constrictions permitting under high flow situations.

It has been confirmed that fines migrate depending on a compromise of pore restriction, rate of flow, and chemical conditions of fluids. Columns can meet different requirements to test different treatments for fines migration damage remediation.

## **ACKNOWLEDGMENT**

The authors wish to thank Petroleos de Venezuela, S.A. and its research branch INTEVEP, S.A., for permission to publish these results..

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Table 1 Salinity Change Program at 1.5 ml/min Rate

Initial Time (min)	Final Time (min)	Salinity (%)	
0.0	5.0	3.0-1.5	
5.0	10.0	1.5-0.0	
K/Ko NaCl	K/Ko BaCl <sub>2</sub>	Injected Vol. (ml)	Salinity (%)
1.00	1.00	0.00	3.00
1.00	1.00	1.50	2.70
1.00	1.00	6.00	1.50
0.93	1.00	7.50	1.35
0.93	0.99	12.00	0.90
0.89	0.99	13.50	0.75
0.86	1.00	15.00	0.60
0.86	1.00	19.50	0.15
0.82	1.00	21.00	0.00
0.71	1.00	22.50	0.00
0.54	0.98	24.00	0.00
0.43	1.00	25.50	0.00
0.40	1.00	27.00	0.00
0.38	1.00	28.50	0.00
0.37	1.00	30.00	0.00
0.36	1.00	31.50	0.00
0.33	1.00	33.00	0.00

Table 2 Flow Rate Change Program at 3% Salinity

Initial Time (min)	Final Time (min)	Flow Rate (ml/min)
0	5	0.5-1.0
5	10	1.0-3.0
10	15	3.0-5.0
15	20	5.0-8.0
Permeability (mD)	Flow Rate (ml/min)	Injected Vol. (min)
2.61	0.60	0.60
2.62	0.70	1.30
2.78	0.80	2.10
2.94	0.90	3.00
2.91	1.00	4.00
3.18	1.40	5.40
3.14	1.80	7.20
2.95	2.20	9.40
3.99	2.60	12.00
4.02	3.00	15.00
4.94	3.40	18.40
5.11	4.20	26.40
4.30	4.60	31.00
4.21	5.00	36.00
4.06	5.60	41.60
4.50	6.20	47.80
4.13	6.80	54.60
3.87	7.40	62.00
3.73	8.00	70.00

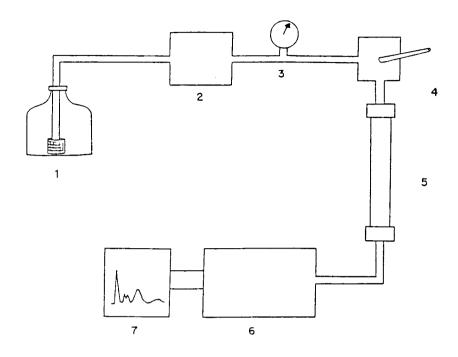


Figure 1 Basic HPLC parts. 1) fluid reservoir, 2) high pressure programmable pump with gradient elution, 3) pressure gauge, 4) injection port, 5) column, 6) detector system, 7) output system.

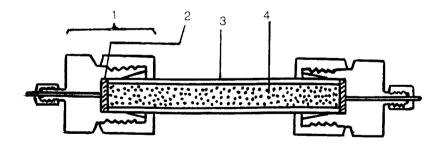


Figure 2 Packed chromatographic column. 1) union with male thread, 2) 0.2  $\mu$ m metal frit, 3) column body (0.7 cm i.d., 9.2 cm length), 4) sandpack with fines.

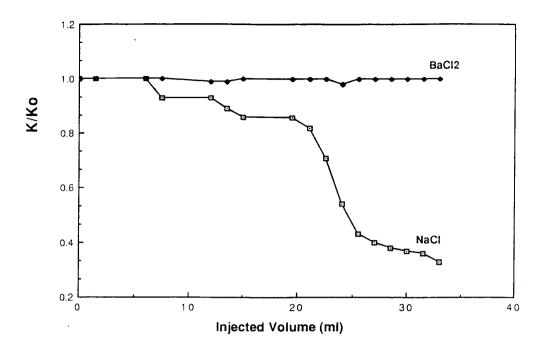


Figure 3 Effect of brine with decreasing salinity on packed column permeability. Salinity values from table 1. Rate flow: 1.5 ml/min.

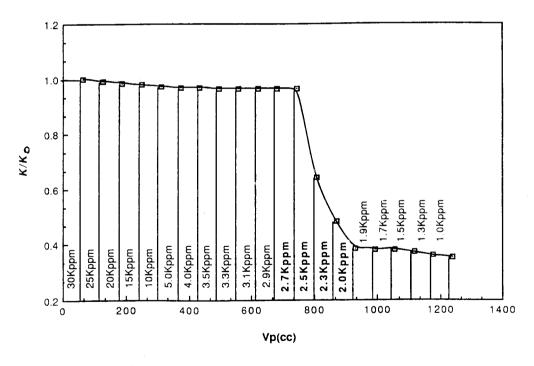


Figure 4 Effect of brine with decreasing salinity on Berea sandstone permeability. Flow rate: 1.5 ml/min.

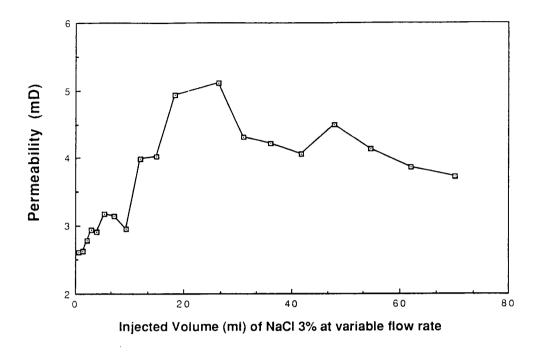


Figure 5 Effect of flow rate change on packed colum with 3% NaCl. Flow rate values from table 2.

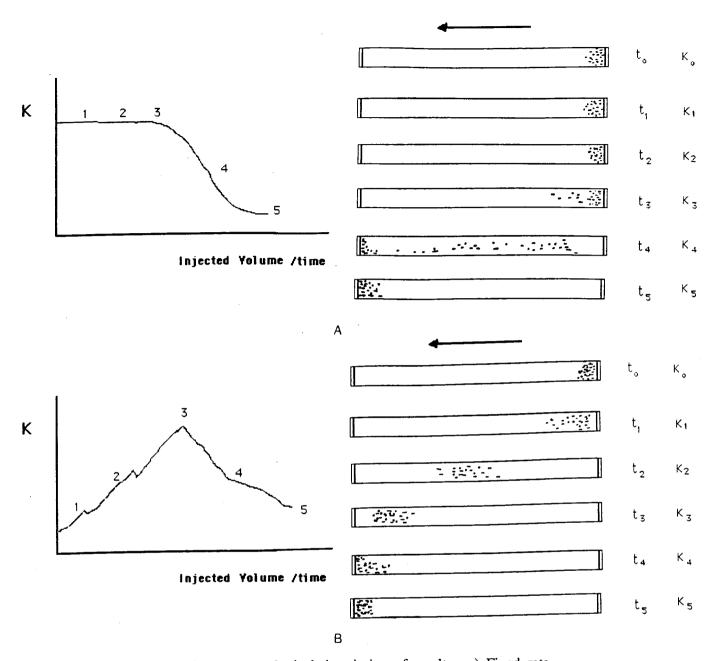


Figure 6 Phenomenological description of results. a) Fixed rate with variable salinity, b) Fixed salinity with variable rate flow.