SCA2003-04: EFFICIENCY OF CLEANING TECHNIQUES FOR OIL AND ESTER BASED MUDS ON UNCONSOLIDATED AND TIGHT SANDS

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Abstract

Wettability is always a key parameter for laboratory core floods. It is well known that cleaning efficiency and wettability restoration quality are key issues when cores are invaded by mud filtrate. This paper illustrates the techniques used in some difficult field cases, where **unconsolidated and tight sands are cored with oil- and ester-based muds.**

Cores were flooded by a series of solvents (toluene, toluene-isopropanol and isopropanol). Gas chromatography as well as mass spectrometry finger-print analyses were conducted on effluents at different stages of the cleaning process, on the crude oil used for wettability restoration as well as on samples of uncleaned and cleaned rock material. These techniques have been used to detect traces of contamination by oil- or ester-based mud filtrate. Complete Amott [1] wettability tests were performed on fresh state cores[1] and on cleaned samples and after wettability restoration. The main results on unconsolidated sands are:

- In all cases, oil- or ester-based mud filtrate was present in the first cleaning effluent, confirming deep core invasion into highly permeable samples.
- Gas chromatography or mass spectrometry does not detect any cases of oil- or ester-based mud filtrate in the last cleaning effluent or in the cleaned core. These results demonstrate that cleaning is efficient.
- No contamination was detected in the crude oil used for wettability restoration.
- The complete Amott tests show that samples are oil-wet[2] or slightly oil-wet at reception, water-wet or neutral after cleaning, and neutral or slightly oil-wet after wettability restoration. This observation is valid for both oil- and ester-based muds from different reservoirs.

The results show that oil- or ester-based mud filtrates influence the wettability of many rocks and confirm the efficiency of the cleaning procedure used. They reveal that the use of fresh-state permeable sand samples leads to erroneous results, with different wettability

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[1] The term "fresh-state" is used for samples taken in a preserved core zone. A fresh-state sample is not cleaned and not dried before the experiment. The term "Native" is sometimes used by other authors with the same or another meaning; it will not be used in this paper.

[2] The terms "oil-wet" and "water-wet" are used in this paper to define a rock preference to oil or water in an oil/water/rock system. These terms are used despite Amott's criticism about their use.

from that of the reservoir. To use fresh state samples, the absence of mud filtrate products must be demonstrated.

Gas chromatography analysis of fresh-state samples of consolidated sand with low permeability (0.1-10 mD) complemented this study. Invasion by oil-based mud was also revealed at the centre of the 10 cm diameter full-size core, which tends to confirm that the fresh-state samples cored with oil-based mud may be deeply invaded, whatever the rock permeability and should be avoided for core flooding experiments.

Introduction

Context and Purpose of the Study

To illustrate the importance of wettability for laboratory core flooding, we shall give a quick overview of water/oil imbibition experiments with immiscible fluids. Many authors, including Hirasaki et al. [5] and Treiber et al. [6], confirmed from such experiments what Cueic [3] demonstrated many years ago: the necessity to reproduce reservoir conditions by replicating reservoir fluids (using reservoir oil and corresponding brine salinity) and restoring reservoir wettability (with sample ageing at reservoir temperature and fluids). The non-observance of this procedure could have a drastic effect on experimental results, such as relative permeabilities and residual oil saturation.

Another obvious parameter is the pore network. Experiments are carried out on reservoir samples. Therefore, it is important to use representative cores. Among the parameters that can affect core properties, are the mud filtrate and core cleaning techniques [1, 3, 4, 5]. Oil-Based Mud (OBM) is often used to core a reservoir. An Ester-Based Mud (EBM) was also used for one of the cores in this study. Trewin [2] has shown that OBM filtrate invades cores. Thus, on the basis of observations made by previous authors, we conclude that **samples cored with oil-based mud may be more oil-wet than the reservoir.**

The work presented in this paper was conducted to assess that risk in Total reservoir studies and was not part of a research program. Specifically, it was added to our conventional procedure and typical Kr program to:

- analyse validity of results in specific fresh-state samples
- analyse the efficiency of our cleaning procedure on unconsolidated sands
- study the influence of ester-based mud, which was new to our experience.

Experimental Work

Amott tests were performed: centrifugation was used for forced drainage and imbibition and Amott Wettability Index (WI) was calculated as defined in Table 1. A complete Amott test was carried out on some samples, which means an Amott test was performed at different stages: upon receipt, after cleaning and after wettability restoration. Experiments were performed on unconsolidated sands under reservoir confining stress using a novel technique.

In addition, geochemical analyses were performed. Some authors [2, 5] have used these techniques to analyse mud invasion, but such a complete analysis on different materials (cleaning effluents, stock tank oil, OBM filtrate, EBM filtrate and rock material), combined with complete Amott tests, to qualitatively analyse mud invasion and cleaning efficiency, has never previously been presented.

Overview of Published Studies

This part highlights results presented in previous papers. Its is not an exhaustive review, but sets out to discuss some important work, which includes many experimental and research studies, conducted very long ago in some cases.

Influence of the Cleaning Procedure

Many authors have studied the effect of cleaning procedure on wettability. Testing procedures were not identical, but in all cases many experiments were performed. The differences are in the types of samples (outcrop, reservoir), the way they are used (once or several times), the wettability evaluation method (Amott test, chromatography, Kr experiment, contact angle, etc.) and the types of solvents used.

Influence on Wettability

Globally, we can say that all authors [1, 3, 4, 5, 7, 8] observed the influence of cleaning on core wettability. Cuiec [3], who described the chemical aspects, made a major contribution in his observations of wettability mechanisms and the difficulty in predicting their quantitative influence. In particular, he pointed out the effect of acidity and basicity.

Influence of Solvents

Those authors also observed the influence of solvents used. For instance, Gant and Anderson [4] showed that toluene is less efficient than toluene followed by flooding with a mixture of toluene/alcohol/1% NH_3OH, and that 1,1,1-trichloroethane makes the sample more oil-wet.

Efficiency of the Cleaning Procedure

It is often thought that cleaning should make the core highly water-wet (WI around +1). However, most papers give totally different results: samples show intermediate wettability after cleaning.

Such results were obtained not only on reservoir cores but also on outcrop samples which are known to be originally highly water-wet. Thus, experiments on outcrop samples are very interesting: they show that the samples, after ageing and cleaning, are not as water-wet as they were before ageing [5].

This is a positive point for the experimental case in a SCAL study, in which the same result is obtained on one reservoir sample:

- Intermediate wettability after cleaning does not prove that cleaning was not efficient.

- Moreover, such a result does demonstrate that samples can retain information on initial wettability even when the cleaning procedure is efficient.

The negative points are that:
- Wettability after cleaning is not sufficient to test the cleaning efficiency.
- The alteration of sample wettability during coring is difficult to evaluate. Indeed, if a cleaning procedure was supposed to make a sample highly water-wet, then when intermediate wettability would be obtained after cleaning, the operator would be sure of sample alteration and its non-representativeness for a SCAL study.

We cannot exclude this negative point by using a more "efficient" cleaning procedure, which would make sample highly water-wet, since we want to maintain reservoir information.

Invasion of Oil-based Mud Filtrate

Trewin [2] showed by quantitative gas chromatography that OBM filtrate invasion exists in all cores but rarely at the heart of the core. The cores studied were more than 4" (inches) in diameter, and the non-invaded heart was at most 2" in diameter, which is not sufficient for experiments carried out on big core samples. Trewin gave advice on how to decrease mud filtrate invasion but this could prove difficult and we wanted to use the core samples even though this preventive method might not work. Therefore, recommendations for coring techniques and core sample size are not sufficient to prevent OBM filtrate invasion in all cases.

Experimental Work

The experimental work is detailed in Table 2. The following sections summarize reservoir and core types and provide details on the experimental procedure.

Reservoir and Core Presentation

The samples used in this study are shown in Table 2 with their field origin, well origin, rock type and experiment types and numbers. Samples from fields A, B and D are unconsolidated fine to coarse sands with a high permeability. However, samples from the B field which are slightly cemented, are less permeable. All samples come from the oil reservoir zone, cored with an oil-based mud for wells in field A and B and ester-based mud for field D. To restore wettability, stock tank oils from wells of corresponding fields were used. These oils and OBM and EBM were analysed. Oil contamination was tested for field C and fresh-state samples from field E were examined for filtrate invasion. The most complete studies were performed for fields A and D.

Wettability Tests

Coring and Preparation

After coring, unconsolidated plugs were set under reservoir stress in cells (length around 50 mm and diameter around 30 mm). Plugs were saturated with brine to first test wettability with fresh-state core samples. Before the second and third wettability tests, the

samples were cleaned and dried. Cleaning consisted of flooding the samples with a series of solvents (toluene, toluene-isopropanol and isopropanol). Samples were dried by nitrogen flooding.

Complete Amott Wettability Test

An Amott wettability test consists of evaluating a wettability index with four experiments on a sample set to Swi: spontaneous imbibition and forced imbibition, spontaneous drainage, forced drainage. Tests were run under oedometric in-situ stress for unconsolidated samples and at laboratory temperature (reservoir temperature was only used for the ageing phase during tests with wettability restoration). Swi was set by centrifugation and forced imbibition and drainage were performed by centrifugation.

In this study, a complete suite of three Amott wettability tests were performed with an oil/water system: on fresh-state samples (no cleaning), on cleaned samples, and on samples after ageing with stock tank oil at Swi at reservoir temperature.

The procedure was not exactly the same as in the Amott test [1]: Amott began his experiments on samples at residual oil saturation; the centrifugation and spontaneous phases were consistently shorter, and the capillary pressure reached was only 1800 times the gravity. For the spontaneous phases, our choice was based on work that demonstrated the importance of imbibition time[3, 9]. For the centrifugation phases, different rotation speeds with production stabilisation were applied to measure capillary pressure.

Centrifugation

Four unconsolidated plugs were placed in a centrifugal machine with four arms; the long axis of each sample (length) was in the horizontal plane, along the arm axis. During the drainage (or imbibition) phase, the machine is in rotation with a velocity that increases step by step. Oil (or water) progressively replaces the brine (oil) in the plug. Observing the displacement of the oil/brine interface allows the measurement of mean expelled volume.

Spontaneous Drainage and Imbibition

Samples in the cell under confining pressure are submerged in water (for spontaneous imbibition) or oil (for spontaneous drainage) until there is no more oil (imbibition) or water (drainage) production. Each phase lasted at least 14 days.

The fluids are recombined brine (30 to 70 eqNaCl g/l) and synthetic oil (Marcol 52).

<u>Geochemical Analyses</u>

LC-GC

LC-GC is a coupled technique between chromatography in liquid phase and chromatography in gas phase. It is the main technique chosen for this study. This technique gives, for each fluid sample analysed, a fingerprint of the saturated hydrocarbons contained in this sample and a fingerprint of the aromatic hydrocarbons. This method also gives a fingerprint of sulphur-aromatic hydrocarbons whenever they are present. Although LC-GC

analysis allows a quantitative approach in the range of saturated hydrocarbons, in this paper we only present qualitative results (internal standards combined with analysed samples nevertheless allow a visual, semi-quantitative evaluation of chromatograms). These chromatography analyses trace the presence of the mud filtrate or original oil in the sampled cleaning effluents and also in the uncleaned and cleaned core material. In addition to cleaning effluents and core material, the same measurements were applied to mud filtrate and stock tank oil.

Other Geochemical Analyses

In some cases, the LC-GC approach was complemented with other analytical methods:

- Iatroscan and HPLC : these are two different kinds of liquid chromatography which have been used to quantify the chemical families in oil, effluents or core extracts samples, in terms of saturated hydrocarbons, aromatic hydrocarbons and polar compounds. Iatroscan is a coupling between thin layer chromatography and flame ionisation detection. HPLC is a high performance liquid chromatography using a stainless column filled with a silica-based solid phase.
- GC-MS is a coupled technique between gas chromatography and mass spectrometry. It enables detection of some compounds that cannot be detected by LC-GC, such as fatty acid methyl esters which are contained in ester-based muds. The sensitivity of this method is far much important than the sensitivity of LC-GC.

Analysed Samples

The samples analysed (numbers and type by field) are summarized in Table 2. The cleaning effluents were taken at different steps of the cleaning process: first toluene effluent, last toluene effluent, last toluene/isopropanol effluent and last isopropanol effluent. The solvents and cleaned core material were also analysed. The core samples used in this effluent analysis were not always the same samples as those used for wettability tests.

Main Results

Figures 1 to 9 (with results for field B) and Figures 10 to 13 (with results for field D for ester-based mud study) are examples of chromatography results that illustrate the conclusions detailed below.

Demonstrated Invasion

Geochemical analyses show significant presence of OBM filtrate in toluene effluents (first cleaning effluents) for samples from wells A3 and B3 (figure 5). This fraction was not available for samples from well B2. For well C, only stock tank oil was analysed in which no contamination was detected. For well D2, toluene first cleaning effluent is highly contaminated by unsaturated and saturated fatty acid esters, which belong to Petrofree and Finagreen based components (figures 10 and 11). Stock tank oil from a similar reservoir in well D1 contained no contamination by esters. **In all cases**, oil- or ester-based mud filtrate was present in the core when analysing first cleaning effluent, confirming **deep core invasion in very permeable samples**.

Cleaning Efficiency

Gas chromatography or mass spectrometry does not detect, in any case, oil- or ester-based mud filtrate and stock tank oil in the last cleaning effluent (figure 7 and 12) and in the cleaned core (figure 9). These results demonstrate that cleaning is efficient.

To be specific, paraffin components were found in cleaned cores (Table 3, Figure 9), which might be due to contamination during the extraction phase when the quantity is large (e.g., sample A3). However, this is in agreement with wettability after cleaning: the WI is really lower than 1. From these results, one interpretation, not checked in this study, can be that after cleaning, samples retain reservoir information, which is completely satisfactory for future SCAL measurements. This phenomenon was already observed with another experiment using XPS analysis (X-ray photoelectron spectroscopy) by Quet et al. [7] who demonstrated that an outcrop sample (Berea) has more carbon components after wettability restoration and after cleaning than before wettability restoration.

Oil and Solvent Quality

No contamination was detected in the crude oil used for wettability restoration. This test is important to ensure the quality of wettability restoration. Solvent quality was also checked.

Wettability Results

Amott wettability test results, presented in Table 4, confirm the cleaning efficiency. The complete Amott tests show that samples are oil-wet – or slightly oil-wet - at receipt, water-wet – or neutral - after cleaning and tend to be neutral - or slightly oil-wet - after wettability restoration.

These results show that oil- and ester-based mud filtrates render core samples more oil-wet than they should be. They confirm that the cleaning procedure used is efficient, but does not induce high water wettability (as confirmed by traces of paraffin observed in the cleaned cores). It shows that the use of fresh-state permeable sand samples leads to erroneous results, as they have different wettability than in the reservoir or after wettability restoration.

Confirmation on Tight Consolidated Sands

Two fresh-state samples of consolidated sand with low permeability (0.1-10 mD) were studied. Those samples were taken at the heart of the 10 cm diameter full-size core. Quantitative gas chromatography analyses were carried out on the extracted effluent by an organical solvent. Quantitative results are not detailed in this paper, but the main result is that extracted effluent was composed of 6 to 10% OBM filtrate. This result corroborates the fact that there is a risk of mud filtrate invasion whatever the rock permeability, and confirms that these samples should not have been used for core flood experiments.

Conclusions

Geochemical analyses and Amott wettability test results were presented to demonstrate that oil- or ester-based mud filtrate invades unconsolidated permeable sands. This invasion renders the core sample highly oil-wet and non-representative of reservoir wettability. Cleaning procedure (toluene, toluene-isopropanol and isopropanol) efficiency was also demonstrated. Similar invasion of tight sand samples has been observed in work that was not detailed here.

This study leads to the following recommendations for core flooding experiments:

- The use of fresh-state samples should be avoided since they lead to erroneous results, as their wettability is different than that of the reservoir.
- If fresh-state samples are used, the core must be checked for absence of mud filtrate products.
- Cleaning procedure efficiency must be checked for each SCAL program, as it was for the present study.

To deal with this issue, a specific program must be set up for each SCAL study. It will be simplified or even more developed than it was for this work, depending on the case.

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Nomenclature

WI: Wettability Index
OBM: Oil-Based Mud
EBM: Ester-Based Mud

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Table 1 - Amott Wettability Index Calculation

Displaced oil volume during spontaneous imbibition **: Va**
Displaced oil volume during forced imbibition **: Vb**
Displaced water volume during spontaneous drainage **: Vc**
Displaced water volume during the second forced drainage **: Vd**

WI	**-1**	**-0.3**	**-0.1**	**0.1**	**0.3**	**1**
WETTABILITY	OIL WET	INTERMEDIATE				WATER WET
		Slightly Oil Wet	NEUTRAL		Slightly Water Wet	

Water Wettability Index **Rw = Va/(Va+Vb)**
Oil Wettability Index **Ro = Vc/(Vc+Vd)**
Wettability Index (WI) **WI = Rw-Ro**

Table 2 – Experimental work presentation

	Field A	**Field B**	**Field C**	**Field D**	**Field E**	**Cleaning effluents**
	Core samples for geochemical analysis : cleaning effluents and core analyses					
Well origin	A3	B2 and B3	-	D2	E	-
Samples origin	2 stacked zones (different facies) selected in the core	Selected in the core	-	2 stacked zones (different facies) selected in preserved core zones	Selected in the core	-
Number of samples	1	3 (B2) + 2 (B3)	-	1	2	-
Number of tested effluents (by sample)	3	1 (B2) ; 3 (B3)	-	3	-	2 for 3 solvents
Rock type	Unconsolidated fine to coarse sands with little clay	Slightly cemented unconsolidated medium sands with little clay	-	Unconsolidated medium to coarse sands with little clay	Consolidated sand	-
Core analysis (see Table 3)	On A3 cleaned core	On B3 cleaned cores	-	On D2 cleaned core	On fresh-state samples	-
	Fluids for geochemical analysis : oil and mud analyses					
Analysed Oil (number and well origin)	1 from A1 - 1 from A2	1 from B1 - 1 from B2	3 from C1	1 from D1	1 from E	-
Type of analysed mud filtrate	Oil	Oil	Oil	Ester for D2	Oil	-
Analysed Mud (number and well origin)	1 from A3	1 from B3	1 from D1	Finagreen analysis	1 from E	-
	Core samples for wettability analysis : Amott wettability tests (see Table 4)					
Well origin	A1/A2/A3	B2	-	D2	-	-
Samples origin	selected in preserved core zones	Selected in the core	-	selected in preserved core zones	-	-
Number of samples	4	4 (3 with tested cleaning effluents)	-	4	-	-
Rock type	idem to geochemical study	idem to geochemical study	-	idem to geochemical study	-	-
Wettability restoration comment	A1/A3 from reservoir 1 (oil A1) ; A2 from reservoir 2 (oil A2)	B2 oil	-	D1 oil	-	-

Table 3 – Cleaned core material analyses

Solvent extraction	Core weight (g)	Extraction weight (mg)	Extractible organic matter (ppm)
Sample from A3	54.12	8	150
Sample from D2	51.54	0.9	20
Sample a from B3	46.38	0.6	10
Sample b from B3	49.04	0.9	20

Table 4 - Amott wettability test results

FIELD A

1st test: fresh state test without cleaning and synthetic oil without ageing

	RESULTS	WELL A1/2/3						
Well	SAMPLE	Va	Vb	Vc	Vd	Rw	Ro	WI
A2	1	0.2	6.52	1	5.62	0.030	0.151	**-0.121**
A2	2	0.3	7.17	0.6	6.46	0.040	0.085	**-0.045**
A1	3	0.25	3.03	0.95	2.67	0.076	0.262	**-0.186**
A3	4	0.4	6.28	5.3	1.39	0.060	0.792	**-0.732**

FIELD A

3rd test: after cleaning with ageing and synthetic oil

Maceration with A2 Stock Tank Oil forA2 samples and with A1 ST Oil for others

	RESULTS	WELL A1/2/3						
Well	SAMPLE	Va	Vb	Vc	Vd	Rw	Ro	WI
A2	1	0.3	6.7	0.8	6.41	0.043	0.111	**-0.068**
A2	2	0.15	6.1	0.3	5.92	0.024	0.048	**-0.024**
A1	3	0.35	3.11	0.8	2.64	0.101	0.233	**-0.131**
A3	4	0.1	6.6	0.9	6	0.015	0.130	**-0.116**

FIELD A

2nd test: after cleaning and synthetic oil without ageing

	RESULTS	WELL A1/2/3						
Well	SAMPLE	Va	Vb	Vc	Vd	Rw	Ro	WI
A2	1	1.2	5.59	0.1	6.08	0.177	0.016	**0.161**
A2	2	1.5	5.27	0.02	6.38	0.221	0.003	**0.218**
A1	3	0.8	2.54	0.02	3.07	0.240	0.006	**0.233**
A3	4	0.5	6.36	0.05	7.08	0.073	0.007	**0.066**

FIELD B

Test after cleaning and synthetic oil without ageing

	RESULTS	WELL B2						
Well	SAMPLE	Va	Vb	Vc	Vd	Rw	Ro	WI
B2	5	2.6	2.57	0.3	3.77	0.503	0.074	**0.429**
B2	6	3.5	3.00	0.6	6.09	0.538	0.090	**0.449**
B2	7	2.5	2.56	0.5	6.61	0.494	0.070	**0.424**
B2	8	3.1	4.14	0.2	5.58	0.428	0.035	**0.394**

FIELD B

Test after cleaning with ageing and synthetic oil

Maceration with B2 stock tank oil

	RESULTS	WELL B2						
Well	SAMPLE	Va	Vb	Vc	Vd	Rw	Ro	WI
B2	5	0.1	4.98	0.45	4.51	0.020	0.091	**-0.071**
B2	6	0.35	6.68	0.35	6.94	0.050	0.048	**0.002**
B2	7	0.01	7.48	0.18	7.77	0.001	0.023	**-0.021**
B2	8	0.15	6.61	0.15	6.38	0.022	0.023	**-0.001**

FIELD D

1st test: fresh state test without cleaning and synthetic oil without ageing

	RESULTS	WELL D2						
Well	SAMPLE	Va	Vb	Vc	Vd	Rw	Ro	WI
D2	9	0.6	7.6	5.8	1.65	0.073	0.779	**-0.705**
D2	10	0.4	7.95	6	1.7	0.048	0.779	**-0.731**
D2	11	0.15	7.51	3.5	3.64	0.020	0.490	**-0.471**
D2	12	0.3	6.82	2.2	4.32	0.042	0.337	**-0.295**

FIELD D

2nd test: after cleaning and synthetic oil without ageing

	RESULTS	WELL D2						
Well	SAMPLE	Va	Vb	Vc	Vd	Rw	Ro	WI
D2	9	0.3	7.27	0.1	6.08	0.040	0.016	**0.023**
D2	10	0.1	6.73	0.25	6.53	0.015	0.037	**-0.022**
D2	11	1.6	5.24	0	5.9	0.234	0.000	**0.234**
D2	12	2.5	3.08	0	5.24	0.448	0.000	**0.448**

Va, Vb, Vc, Vd : volume in cm3

FIELD D

3rd test after cleaning with ageing and synthetic oil

Maceration with D1 stock tank oil

	RESULTS	WELL D2						
Well	SAMPLE	Va	Vb	Vc	Vd	Rw	Ro	WI
D2	9	0.05	7.75	0.45	6.59	0.006	0.064	**-0.058**
D2	10	0.02	7.79	0.6	7.06	0.003	0.078	**-0.076**
D2	11	0.02	7.14	0.5	6.5	0.003	0.071	**-0.069**
D2	12	0.05	5.88	0.45	5.56	0.008	0.075	**-0.066**

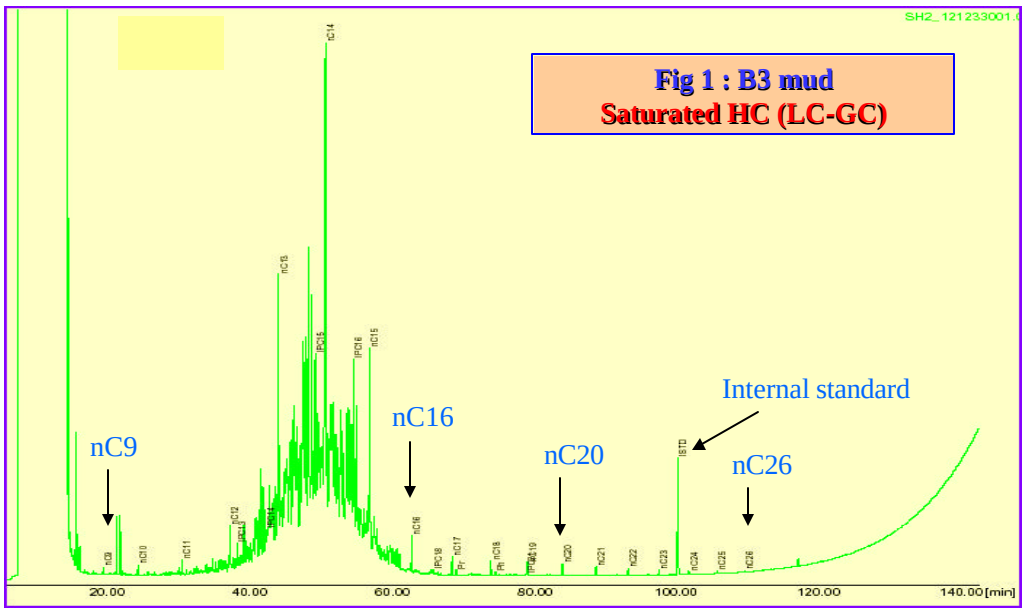


Figure 1 :B3 mud (LC-GC - Saturated HC)

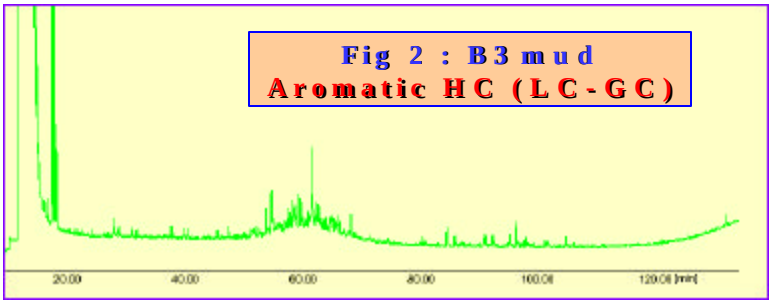


Figure 2 : B3 mud (LC-GC - Aromatic HC)

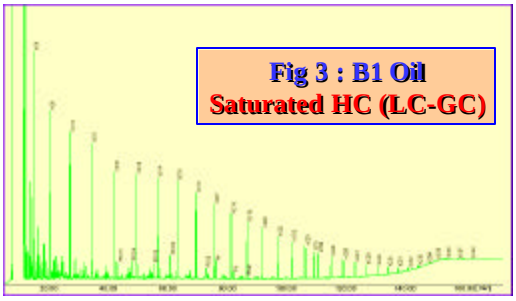


Figure 3: B1 Oil (LC-GC - Saturated HC)

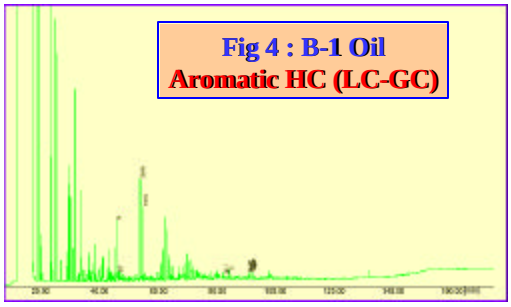


Figure 4: B-1 Oil (LC-GC - Aromatic HC)

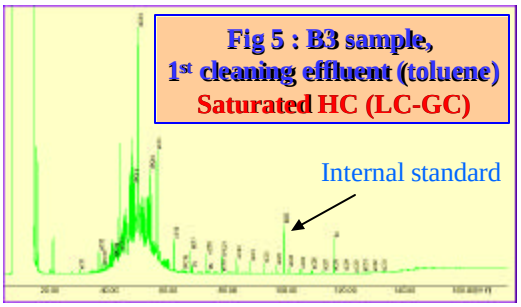


Figure 5: 1st effluent (Saturated HC)

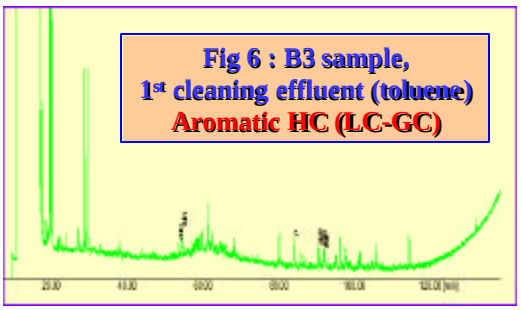


Figure 6: 1st effluent (Aromatic HC)

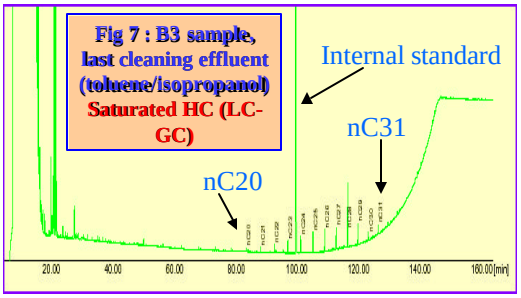


Figure 7: last effluent (Saturated HC)

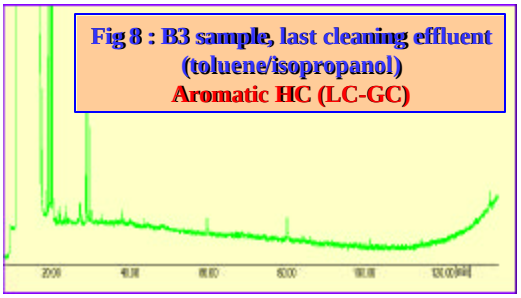


Figure 8: last effluent (Aromatic HC)

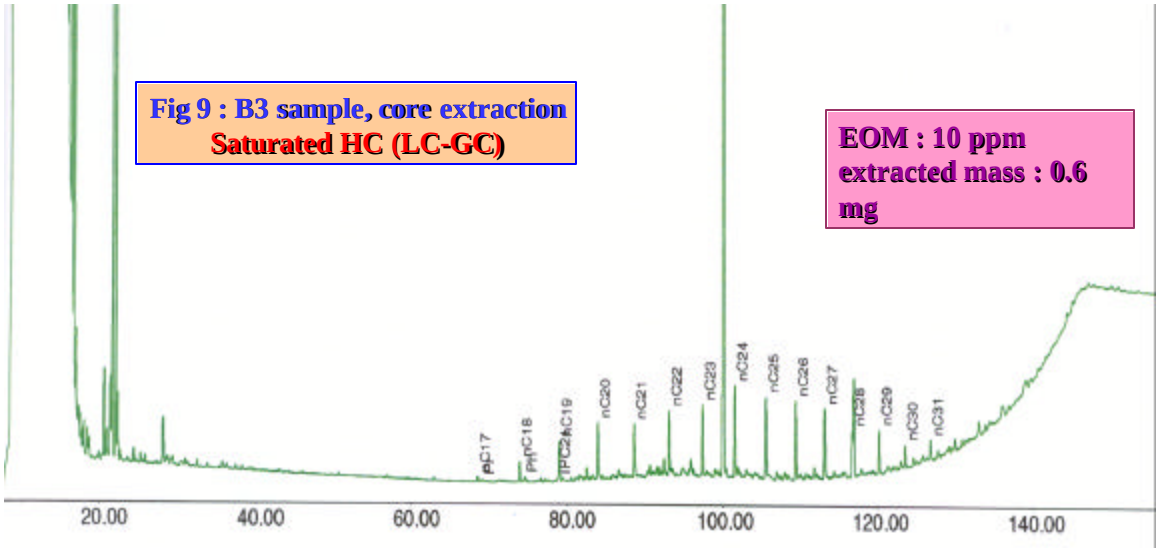
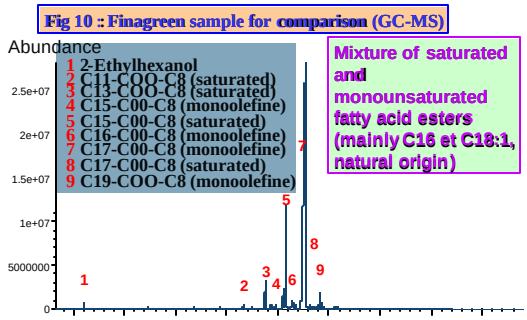


Figure 9: B3 sample, core extraction (LC-GC - Saturated HC)



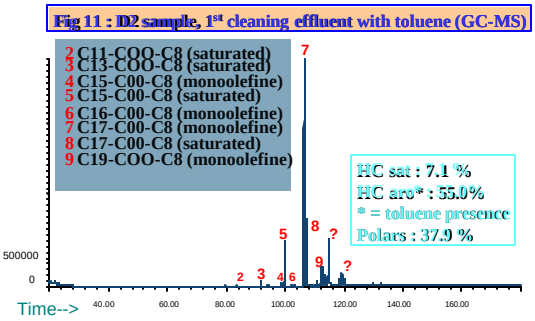
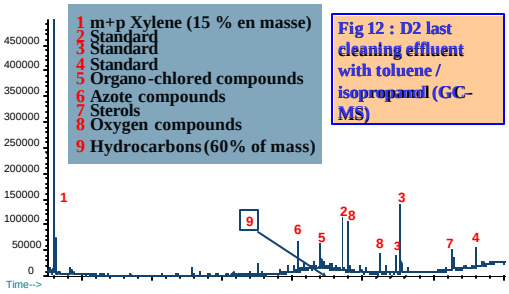


Figure 10: Finagreen (Ester) sample for comparison

Figure 11: D2, 1st cleaning effluent



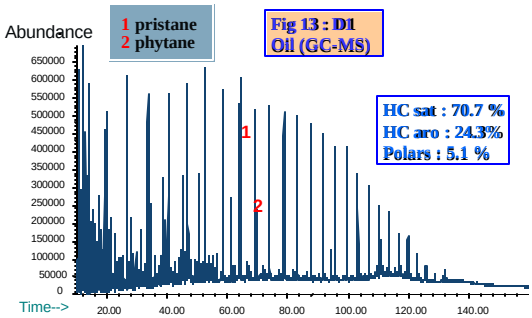


Figure 12: D2 last cleaning effluent (GC-MS)

Figure 13: D1 Oil (GC-MS)