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DETERMINATION OF MUD INVASION CHARACTERISTICS OF SANDSTONE RESERVOIRS USING A COMBINATION OF ADVANCED CORE ANALYSIS TECHNIQUES

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ABSTRACT

A combination of novel petro-physical methods has been used for an integrated investigation of the saturation conditions and characteristics of saturating fluids near the well-bore in sandstone reservoirs. The applied methods include Magnetic Resonance Imaging (MRI), Magnetic Resonance Relaxometry (NMR), Ultra-centrifuge and Gas Chromatography (GC) in combination with Mass-Spectrometry. Routine petro-physical analysis has also been done. The recovered results were used as a basis for the investigation of different objectives. One such objective was the demonstration of NMR logging tool applicability for the study of the invasion. The following subjects were investigated:

- Similarities and differences of NMR instrument results from imaging and relaxometry.
- The three dimensional porous structure of the core samples.
- The effective porosity determination by the NMR method and the evaluation of different models for permeability estimation under invaded zone conditions.
- The informative NMR parameters for the identification of the saturation conditions near the well-bore.
- The wettability alteration of porous media as a result of different kinds of treatment.
- The influence of porous structure and lithology on the fluids interaction with pore surface.
- The physical-chemical parameters of oil in the porous media and in the bulk volume before and after displacement.

The results of the performed project can be used as a guideline for the determination of native state reservoir characteristics and the estimation of the saturation conditions near the wellbore during the different drilling/completion/production stages.

INTRODUCTION

The investigation of mud invasion effects by the Low Field NMR method under laboratory and field conditions were demonstrated in recent publications[1,2]. In some publications[3,4], attempts to investigate core samples with different NMR techniques for better characterization of the physical-chemical properties of the fluid saturated porous media were demonstrated. The application of the two different NMR techniques gives the possibility for deeper investigation of the porous structure, wall relaxation effects and diffusion-in-gradient effects[5,6].

A combination of the bench-top Nuclear Magnetic Resonance (NMR) relaxometer and high field Magnetic Resonance Spectrometer/Imager (MRI) is used to assist the NMR logging tools used in Canadian oil fields. The combination of these novel techniques form the basis of the different interpretation methods that were applied as support to a wide range of solutions which can be recovered by NMR.

EXPERIMENTAL

The presented study was prepared using a collection of core samples from terregineous deposits with a wide range of properties (Table 1). All samples were tested in the NMR without any initial information or core descriptions to the laboratories (PRI and TIPM). The NMR testing was done using both low and high field NMR devices. Cores were investigated under different saturation conditions which simulate different states of the porous space in near well-bore zone (native or invaded).

A formation brine was reconstituted from water analysis data. The waxy crude oil from the deposits under study was acquired and filtered at specified temperature and pressure. Six plugs specified by the operator (see Table 1) were cleaned using toluene, methanol/acetone and n-octane. The dimensions of each plug were measured and the bulk volume was calculated. Following saturation, the bulk volume was also measured using the Archimedes principle. Each plug was then saturated with brine. The porosity and brine permeability were measured. Each plug was tested in the NMR at 100 % H_2O saturation. The MRI (high field) measurements for these saturation conditions were carried out for just three plugs. The plugs were then placed in an ultracentrifuge and were spun at temperature and at 4,000 RPM. Again, the plugs were scanned in the low (relaxometer) and high (spectrometer) field NMR. The NMR spectra for displaced oil samples were recovered from each core by both devices.

The next step was displacement of the core fluids by the drilling mud in core holders. The tests were performed at 80^{o}C, at 1000 psi overburden pressure and at atmospheric pore pressure. The drilling mud consisted of IPAR-3 fluid and a 20%wt. $CaCl_2$ brine at a ratio of 7/3 by volume. At the end of the experiment, the plugs were once again tested with

both NMR devices. The NMR characteristics of mixed displaced fluid (mud+oil) were measured with the NMR relaxometer. All liquids were kept for more than three months as bulk volume liquids in the sealed flasks prior to testing. In each sample the hydrocarbon components settled under gravity. All samples were then separated with a syringe. The viscosity of each sample was measured using conventional methods. Additional fluid characterization was performed using Gas Chromatography. Four fluid samples were investigated: the initial filtered oil, a mixture of initial treated oil and IPAR-3 (50:50 by weight) and the oil-mud mixtures displaced from cores 8A and 70 (see Table 1 and Table 5). Then all plugs were cleaned with toluene, methanol/acetone and n-octane. After this, the plugs were saturated with brine, tested in the low field NMR, spun under air at 4000 RPM for 4 hours and tested in the NMR relaxometer again.

The low field NMR study was carried out with Numar Corespec™-1000 bench-top relaxometer that emulates the performance of the Numar (now owned by Halliburton) MRIL Tool that is utilized in Canada by Computalog and Western Atlas. The equipment is installed and tuned at the TIPM Laboratory and operates at a frequency of 1 MHz (0.024 Tesla). For all measurements CPMG sequences were applied with inter-echo times 0.5 ms and 1.2 ms. All samples at all saturation conditions were tested in the NMR at room temperature and ambient pressure. All decay data are translated into NMR spectra using algorithms developed in-house and NUMAR standard analysis packages[17] that are included with the relaxometer. The high field NMR imaging and relaxation measurements were performed at 100 MHz (2.35 Tesla). An inter-echo time of 0.6 ms was used for the relaxation decay curves and an imaging echo time of 5.0 ms was used for the scanning. Lengthwise and radial full core plug images were obtained with no “slice selection”. All high field NMR measurement were done at the Petroleum Recovery Institute (PRI). All data processing was performed with PRI in-house software.

Porosity, permeability, irreducible water saturation (S_{wirr}) and oil viscosity were determined using direct measurements and low field NMR data recovered under different saturation conditions.

RESULTS AND DISCUSSION

Figure 1 shows an example of comparison of the NMR spectra for core sample 8A recovered with low and high field NMR techniques. All spectra from high field NMR are shifted to shorter T_2 terms. In Table 2, the geometric T_2 means are calculated from low and high NMR spectra. All values from the high field NNR are lower than the equivalent parameters from the low field NMR measurements. This difference is expected, because internal gradients in the porous media arise[5] under the applied high magnetic field. The internal gradients (G) in the same porous media would be different by more than 10 times under high (85-100 MHz ^{1}H Larmor frequency) and low (1.0-2.1 MHz ^{1}H Larmor frequency) magnetic fields[5]. This difference strongly influences the estimated core samples characteristics[5]. For the purposes of this research, which was performed as a support for

the low field logging tool data interpretation[10], low field NMR results were used for quantitative analysis and the high field NMR technique was applied for reconstruction of the 3-D pore space.

Figure 2 shows images of four of the plugs tested. Figure 2a shows of the sample 20A structure. A diverse pore size distribution exists for this sample. Part of the sample is laminated and part of the sample does not have laminations. The image from Sample 70 (Figure 2b) shows large diagonal laminations. The majority of the pore space appears in sample 27A (Figure 2c) to be composed of large pores. There appears to be some bedding structure in the middle of the sample. The majority of pore space in sample 8A (Figure 2d) appears to be composed of small pores, although large pores appear as bright spots. These structural differences can not be identified during the visual lithological description.

Indication of Saturation Conditions

Figure 1 and Figure 3(a, b) show typical NMR spectra for samples with different pore structure and lithology. The NMR spectra for sample 70 (Figure 3a) and sample 8A (Figure 3B) show that the 100% brine saturated sample and the mud invaded core. Similarities and differences can be identified. The geometric means of the T_2 values can be used as a quantitative parameter for the identification of the saturation conditions between natural oil saturated zones and zones after mud invasion for all studied core samples (see Table 1). The combination of NMR log with any conventional resistivity logs with the same radial depth of investigation will solve the problem of saturation determination.

Total Porosity

The initial amplitude of the NMR waveform is proportional to hydrogen density[5,6]. The NMR porosity is usually determined under brine/water saturation conditions. This porosity is different than the log measured porosity in situ at the limits of oil/gas saturated zones or at the invaded zones. In the presented project, the NMR porosity was determined at two saturation conditions; 100% brine saturation and saturation after water displacement by oil. The normalization according to liquid hydrogen indices was done and verified against mass balance. This procedure was carried out for calculation of water-filled and oil-filled volume of pores. This is possible because the signal from hydrocarbons' hydrogen and from water's hydrogen in the porous media can be precisely divided[12]. The two NMR porosity predictions were determined with precision 0.3% porosity units. The total hydrogen signals from cores and known standards[5] were used for the porosity determinations. If the hydrogen indeces for saturating liquids in the invaded zone are strongly different, the NMR total porosity under these conditions can not be determined correctly. The mud-oil solubility[12] in the invaded zone also can cause deviations in NMR porosity predictions. The fluid saturation in the invaded zone can not be simulated correctly and leads to erroneous estimation of the total porosity by the NMR log tool.

Permeability

There are three widely used estimators of permeability (k_{NMR}) from NMR logging: (1)[15] $k_{NMR}= \phi^4 / S_{wirr}^2$, (2)[5-8] $k_{NMR}= C\phi^4 / (T_2)_{mean}^2$ and (3)[1,9,10] $k_{NMR}= ((\phi/10)^4 * (FFI/BVI)^2)$. In Table 3, permeability values calculated by using above the described models and experimentally derived permeability values are presented. Methods for NMR permeability calculations use a "bundle of tubes" approach or empirical relations that we believe are simplistic.
All these "universal" estimators are based on the NMR signal from the 100 % brine saturated cores. Permeability comparisons thus must be made with brine permeability values to avoid discrepancies due to gas slippage (Klinkenberg effect). "Routine core analysis" air permeability values usually obtained on single flow rate vs. single pressure drop measurements, have little use for modelling purposes. Brine permeability measurements reported here (Table 1) are based on measurements of at least four points (different flow rates and corresponding pressure drops) and permeability is obtained through linear regression of Darcy's Law. Moreover, NMR comparisons of permeability should be made only when the same hydrogen-bearing fluid in both NMR and routine core tests is used. Additional errors can be appear from the non-correct estimation of the model parameters. S_{wirr} strongly depends on pore space geometry, pore surface wettability and saturation history. Experience[1,5,6] shows that for each reservoir and particularly for different saturation zones the T_2 cutoff for the FFI/BVI estimation has to be determined by specific experimental studies. The data from Table 3 show that the recommended[1,5,6] T_2 cutoff gives erroneous results for the core samples that were investigated in this work. The data from Table 3 show that k_{NMR} with $k_{measured}$ data in the best conformity provided by the FFI/BVI model. This result took place when FFI/BVI data were compared with air permeability measurements. However, such a comparison contradicts the different physics associated with permeability measurements. Moreover, this estimator is not general, as shown next.

The verification of the commonly used FFI/BVI model was done by using a set of data from terrigeneous oil and gas reservoirs with significantly different lithological composition, pore structure, pore surface wettability, etc[18,19]. Figures 4(a, b, c) show the characteristics of the test data and the permeability estimations with the FFI/BVI model. The difference in some estimated data is more than one order of magnitude and may be attributed to heterogeneity of deposits, saturation conditions (oil and oil+mud), differences in NMR parameters of water and hydrocarbons, differences in pore surface wettability in the water saturated zones and pay zones of the reservoirs, differences in S_{wirr} values in extracted and native state cores, and changes in oil composition. Models that are tailored to particular rock geometry (pore size distribution and pore connectivity) and fluid conditions in the pore space (saturation conditions, current wettability) should be more appropriate for reservoir permeability predictions with NMR.

Wettability alteration and chromatographic separation of the oil in the porous media

Advanced techniques can be applied for the estimation of the physico-chemical composition of oil in-situ and the investigation of the different effects on the oil/water/rock contacts in the porous media[1,12,13]. Chromatographic separation of oil[13,14] can take place during the flow of oils through porous media especially for heavy oils (viscosity more than 50 cP) with high content of polar components[13,14]. Additional adsorption of polar components of oils on the pore surface and is associated with wettability alteration. Compositional oil changes can be verified by NMR and by routine viscosity measurements of the initial oil and the oil after displacement[13]. In this project, core samples and fluids were investigated by NMR and liquids were studied by gas chromatography.

The maximum differences between the NMR spectrum of initial oil and spectra of oil/mud mixtures after oil displacement by mud from samples 8A and 70 were found. These two plugs are different in mineralogy of cement, pore space topology, porosity, permeability and irreducible saturation. Both cores were treated with the same procedures (cleaning, brine/oil/mud saturation, drainage/imbibition stages, temperature, aging time) and with the same initial quality of liquids (brine/oil/mud). Additional adsorption and wettability alteration in both core samples can be explained by:

- Penetration of polar oil components through water films, as observed for oils enriched by asphaltenes (~ 9-10 % wt.)[20,21]. The amount of polar components in the oil used is ~25% (~11% of asphaltenes).
- Low irreducible water content (<6%) in both cores is a positive condition for oil/surface interactions[14,20].
- Possible acid/base interaction that control surface charge at oil/water and solid /water interfaces[24]. According to temperature dependent and solvent dependent adhesion our data are in the region where adhesion is always observed (Brine pH=5.6; concentration of $[Na^{+2}]$ is 0.89M).
- Ion binding or specific interaction between charged sites and higher valency ions (Ca^{2+} and Mg^{2+}) present in the brine (0.34% $CaCl_2$; 0.14% $MgSO_4$)[21,24].
- Adsorption of the hydrocarbon components with higher molecular weight (C_{20+}=25.9%)[20,19,22].

Higher hydrocarbons' adsorption on the pore surface of core 8A is expected due to:

Difference in mineralogy of cement. The cement of this core is dolomite. Dolomitization is characterized by decreasing of the volume of rock matrix (up to 10-12% of initial volume[23]). Pore accessibly largely influences wettability of carbonates. Micropores are water-wet while mesopores are found to be oil-wet[25]. In carbonates, additional chemical adsorption on the surface of the new chemical components (as naphtenic calcium) is possible.

Topology of the porous structure. As shown by the MRI imaging, sample 70 seems to be more homogeneous than sample 8A. The structure of sample 8A is assumed to be a combination of small pores and mesopores. During oil invasion, oil first invades the larger pores of the initially water-wet pore space, while the smaller ones may remain oil-free. Aging may then cause adsorption of oil polar components onto possible exposed pore surfaces either directly or through a remaining thin film of brine. These surfaces then become oil-wet.

Irreducible saturation in core 8A is lower than in core 70, thus the pore surface of this core more sensitive for oil chemistry than pore surface of plug 70. Table 4 shows the difference between the S_{wirr} values predicted from NMR cutoff points for the brine and for the oil/mud saturation conditions.

Adsorption and wettability alteration can explain changes in the NMR spectra of cores. In order to use NMR as a quantitative core analysis tool, the following observations have been made[14]: (1) H^1 nuclei from water and oil do not interact, (2) H^1 nuclei of hydrocarbons from the different chemical groups (saturates, aromatic etc.) weakly interact (possibly on films) and H^1 nuclei of hydrocarbons from the same chemical groups strongly interact, (3) oil/solid and water/solid interactions affect the NMR relaxation and shift T_2 spectra to shorter terms.

In Figure 5b the longest T_2 terms in the invaded zone NMR spectrum (sample 8A) are shorter than in the spectrum for the same conditions for sample 70 (Figure 5). This result can not be correlated to a diffusion effect in sample 8A but rather due to the mesopores of this sample. Approximately 40% of the NMR amplitude (invaded zone, plug 8A) is shorter then the amplitude at Sw=100%. This result for sample 8A can be interpreted as an effect of the additional adsorption of the polar non-soluble hydrocarbon components (resins and asphaltenes) on the surface of large pores which are constructed with dolomite cement, i.e. [oil+mud]/[polar components]/[solid] interaction (see Table 1, Figure 2d). For sample 70, the difference in long T_2 terms of the spectra is not essential in the frame of the fitting precision. 30% of total amplitude according to T_2 values is equivalent of T_2 values from the NMR spectra for the bulk volume of same displaced liquid. This is an indication that the [oil+mud] liquid fills the pores with thick water films without collapsing these films, without interacting with the pore surface and without significant adsorption on the pore surface. This core still is predominantly water-wet after treatment by oil followed by a mud treatment. If additional adsorption took place in the porous media and the oil changed chromatographically, we can expect a different composition of the oil in the [oil+mud] mixture after oil displacement by mud from different plugs (8A&70). It is known[16], that resins and asphaltenes are not soluble in kerosene and play a more active role in the adsorption process. If so, the displaced oil must have a reduced concentration of polar components.

GC analysis data (see Table 5) show the differences in oil composition for displaced liquids from sample 8A and 70. The small amount of polar non-soluble oil components indicate that in the pore space of sample 8A additional adsorption took place. Different results were obtained form GC analysis of the [mud+oil] mixture displayed form sample 70. Normalized data for the amount of C_{20+} components in the displaced mixtures show that the amount of these components in mix-70 is 1.44 times higher than in mix-8A.

For the separate identification of these effects, NMR analysis of the displaced liquid after mud invasion was performed. Figure 5 shows the two NMR spectra of oil - mud mixtures displaced from samples 8A and 70. The shortest terms in the spectrum for 8A mud oil sample(Figure 5a) are negligible. That can be correlated with the small amount of the heavy hydrocarbon components (resins and asphaltenes).

CONCLUSIONS

The laboratory NMR work is being performed with the aim of calibrating the NMR logging tools for use in open hole logging. The term "calibration" is quite general and implies determining the precision and accuracy of the NMR signal with respect to a given parameter. The data presented address the following points with respect to such calibrations:

- In-situ saturation conditions can be obtained by NMR logging using the T_2 geometric means.
- Total NMR signal correlates well with total porosity for water and oil saturated zones.
- The NMR porosity estimation is difficult in mud invaded zones without any information about brine/oil/mud mixture composition.
- Absolute permeability derived from NMR logs using known models is only a preliminary estimation.
- The cutoff of T_2 values for S_{wirr} estimation has to be independent of the brine/oil/mud saturation conditions.
- Determination of movable vs. non-movable water usually can be done if the air-brine S_{wi} signal is obtained. Similar claims can be made to determine movable and non-movable hydrocarbon. The "non-movable" fluid component is generally associated with the low T_2 component of a spectra derived from an oil saturated core, caused by surface adsorption, jammed droplets or other surface related phenomena. Such interpretation also requires the assumption of a pore level model to describe the reservoir.
- Combination of the NMR and Gas Chromatography methods is an additional tool for determining the adsorption of the polar oil components at the pore surface.

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Table1: Characteristics of the core samples

Sample	Porosity (helium) %	Porosity (weight) %	Porosity (NMR) %	S_{wirr} (centrif.) %	Perm (air) mD	Perm (brine) mD
40	20.8	20.4	19.7	0.90	1920.0	1696.2
70	24.5	22.3	21.6	5.6	723.0	203.6
65A	19.4	18.9	18.8	11.10	145.4	8.6
27A	20.7	20.4	20.0	9.80	620.7	237.9
20A	16.8	15.7	15.3	1.30	275.9	70.0
8A	11.0	10.9	10.4	3.70	46.2	34.2

Table 2: Geometric means of the transverse relaxation times (T_2) recovered with low and high field NMR spectrometers (different saturation conditions)

Sample	100 % Brine Saturated		Oil/Swirr Saturation		Invaded Zone	
	HIGH	LOW	HIGH	LOW	HIGH_	LOW
8A	13.1	149.2	11.6	59.5	14.8	162.7
20A	65.0	113.9	28.6	67.3	40.3	153.7
27A	52.0	251.7	58.7	73.7	84.5	221.6
40	N/A	123.0	33.6	60.5	58.3	154.1
65A	N/A	102.0	39.0	62.2	58.4	143.8
70	N/A	129.2	36.7	67.1	60.7	174.0

Table 3: Permeability estimation

	Model1: T2_AVER			Model2: T2_FFI/BVI					Timur: Model3
Sample	S_w=1.0	S_{oil}+S_{wirr}	Invaded	S_w=1.0	S_{oil}+S_{wirr}	Invaded	Perm (air)	Perm (brine)	Sw=1.0
27a	453.23	41.32	352.78	1234.8	293.7	1206.88	620.7	237.9	1537.8
40	107.18	82.62	156.31	481.18	336.24	1036.35	1920	1696.2	N/A
65a	60.21	21.46	87.91	346.2	159.8	394.56	145.4	8.6	481.9
8a	7.19	1.71	46.38	25.34	13.7	84.97	46.2	34.2	37.3
70	121.67	46.32	293.7	928.3	443.96	2784.7	723	203.6	1230.6
20a	33.07	11.74	60.83	214.27	107.1	272.8	275.9	70	292.0

Table 4: Estimation of the irreducible water saturation with different T_2 cutoff values (relaxometer)

Sample	S_{wirr}-wt	NMR_S_{WIRR}	Cutoff_10ms	Cutoff_22.6 ms	Cutoff_22.6 ms	Cutoff_22.6 ms
	Centrifuge	Centrifuge	Brine	Brine	Oil+S_{wirr}	Invaded zone
	%	%	%	%	%	%
8a	3.7	8.2	3.4	17.7	22.6	10.5
65a	11.1	11.7	7.2	16.1	22.0	15.3
27a	9.8	11.2	5.5	10.2	18.9	10.3
20a	1.3	0.4	8.4	13.7	18.5	12.4
70	5.6	9.9	9.3	13.3	18.1	8.1

Table 5: CG-MS results for oil-mud mixtures

Sample	Sample	Saturates	Aromatics	Polar A	Polar B
	name	wt %	wt %	wt %	wt %
1	Initial oil	38	36	18	8
2	50-50	53	27	15	5
3	From 70	58	20	14	7
4	From 8A	80	9	6	5

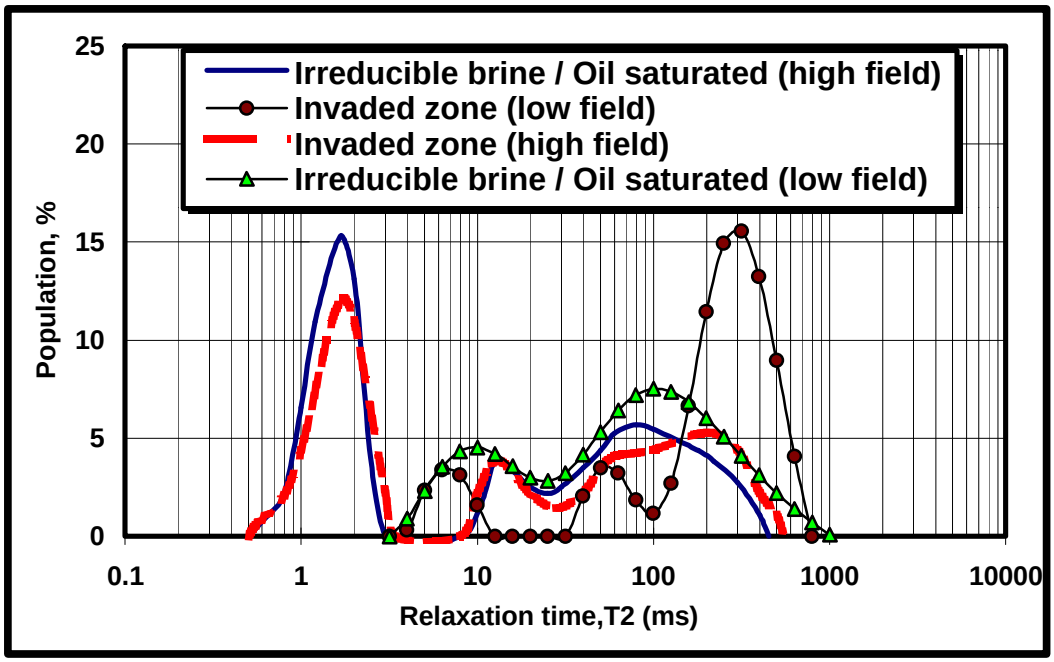

Figure 1: NMR spectra for sample 8A recovered by low and high field NMR techniques

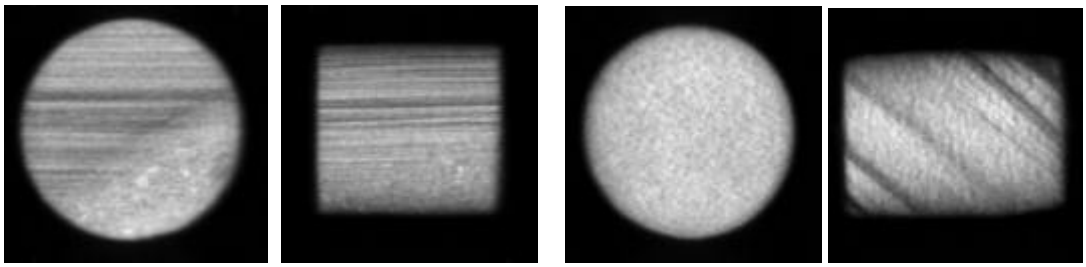

Figure 2a: Radial and Lengthwise NMR Images (Full Volume) for Sample 20A

Figure 2b: Radial and Lengthwise NMR Images (Full Volume) for Sample 70

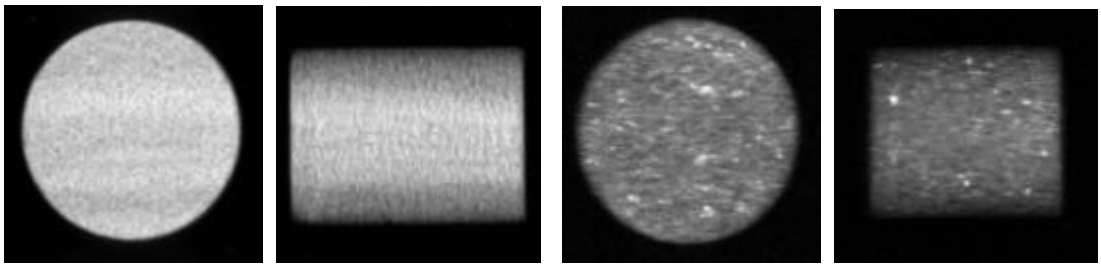

Figure 2c: Radial and Lengthwise NMR (Full Volume) for Sample 27A

Figure 2d: Radial and Lengthwise NMR Images Images (Full Volume) for Sample 8A

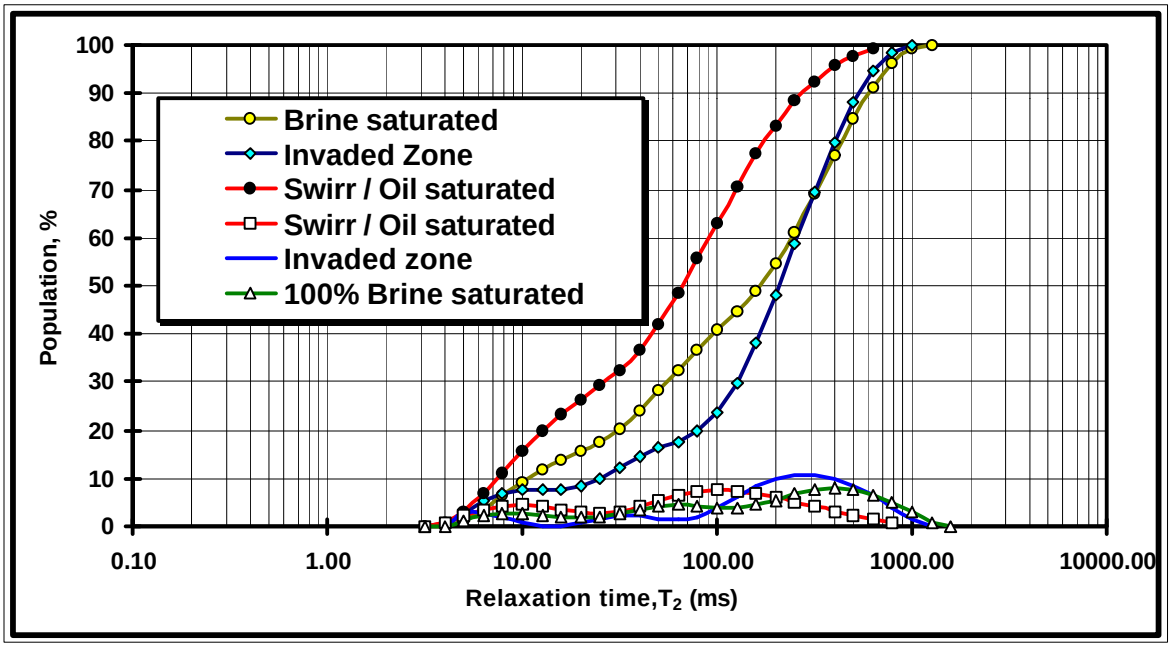

Figure 3a: NMR Spectra for sample 70 (low field NMR)

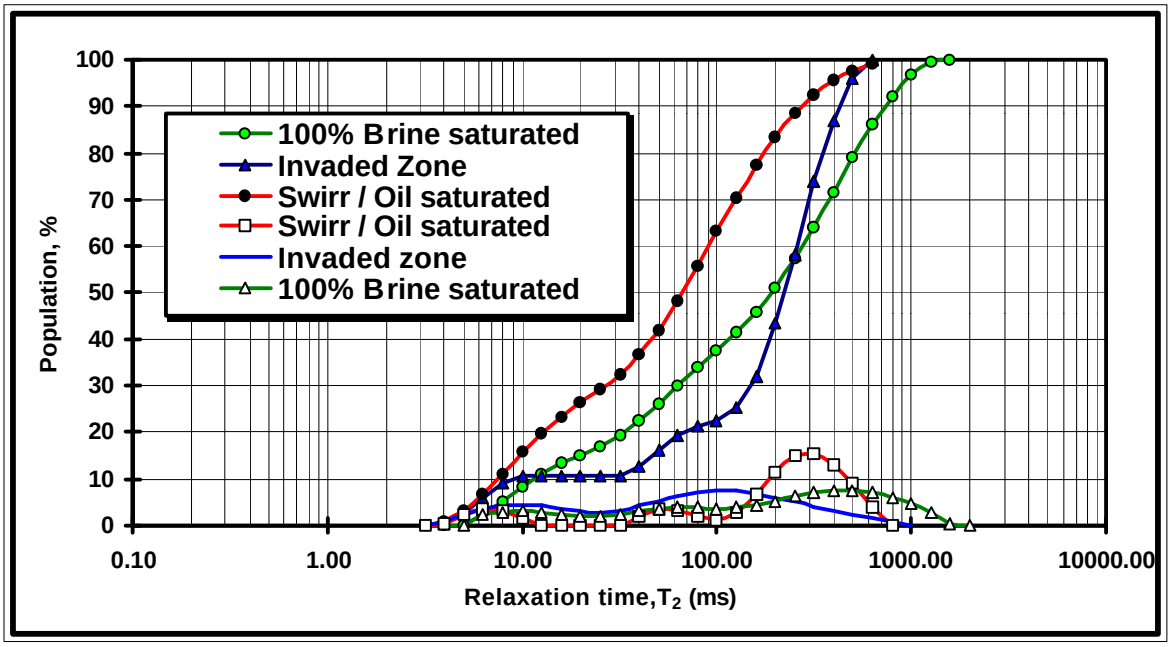

Figure 3b: NMR Spectra for sample 8A (low field NMR)

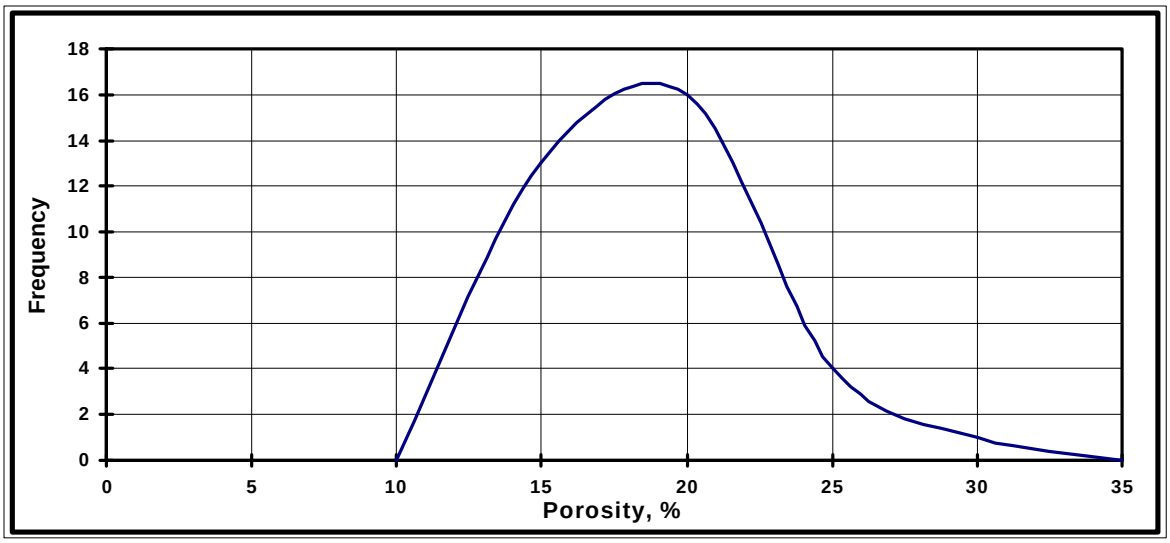

Figure 4a: Porosity distribution for test data which was applied for FFI/BVI model verification

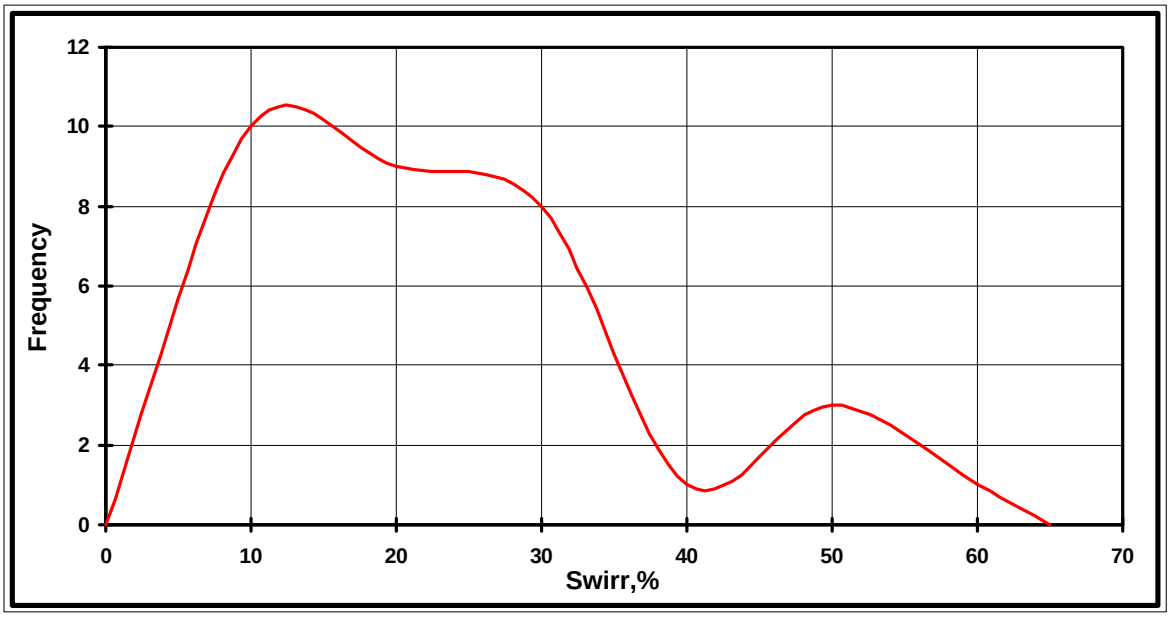

Figure 4b: The S_{wirr} distribution for test data which was applied for FFI/BVI model verification

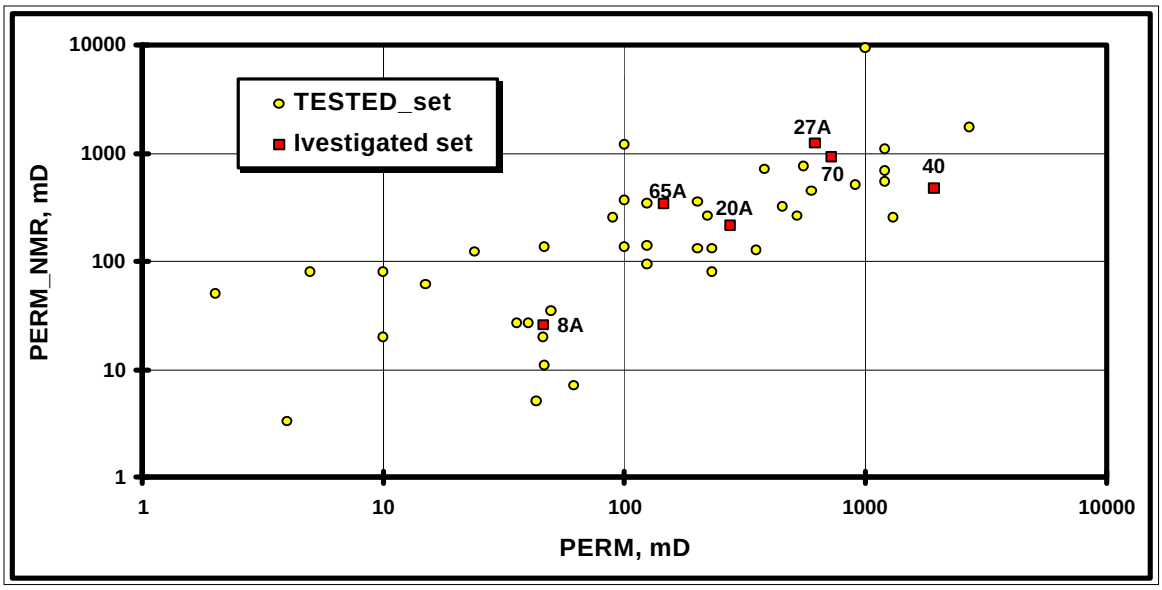

Figure 4c: Results of the FFI/BVI model verification with investigated and test data usage

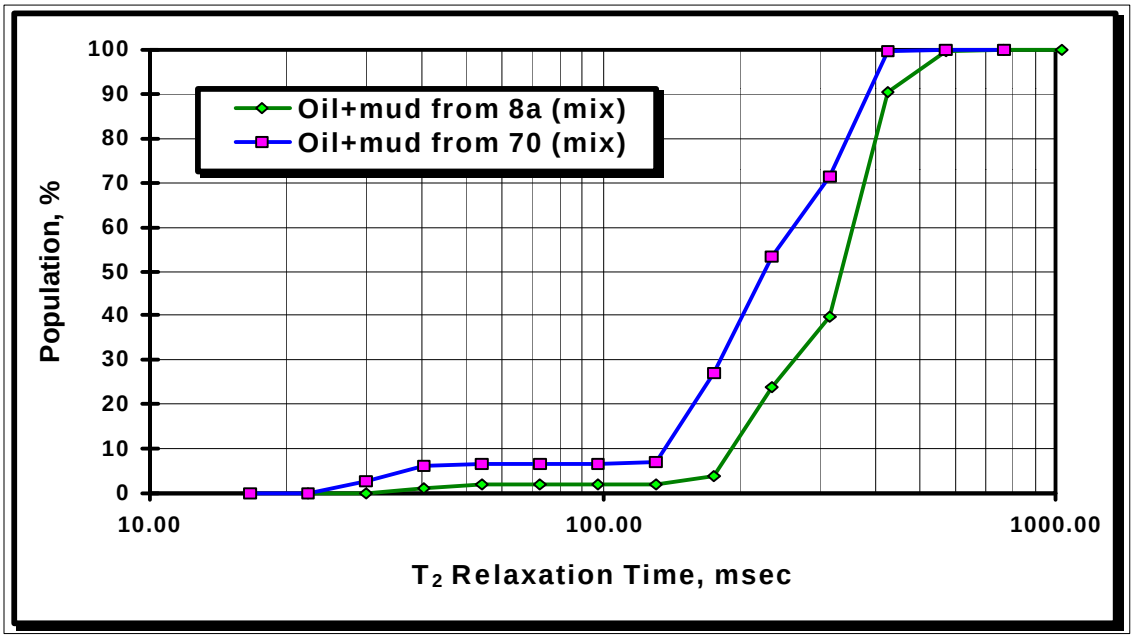

Figure 5: The NMR spectra for oil+mud samples (after mud invasion)