Determination of Oil and Water Compositions of Oil/Water Emulsions Using Low Field NMR Relaxometry

K. ALLSOPP, I. WRIGHT, D. LASTOCKIN, K. MIROTCHNIK
Tomographic Imaging and Porous Media Laboratory (TIPM)

A. KANTZAS
TIPM Laboratory and University of Calgary

Abstract

During production operations in heavy oil and bitumen formations where thermal recovery methods are applied, the fluids produced are often in the form of emulsions. This is also true in non-thermal recovery methods whenever oil and water are co-produced, but to a lower degree of severity. Conventional flow measuring devices are capable of measuring oil and water streams when they are segregated, but they fail when oil-in-water or water-in-oil emulsions form. Conventional methods are also not reliable when there are solids flowing in the stream.

Low field NMR relaxometry was successfully tested as a tool for accurately measuring the oil and water content of such streams with and without emulsions present in the samples. The method was proved to be at least as good as conventional extraction methods (i.e., Dean-Stark). The technology was tested with both artificially and naturally occurring emulsified streams with accuracy better than 96%. This extremely encouraging result led to the design of an online NMR relaxometer for oil/water stream measurements under the conditions encountered in the production of heavy oil and bitumen.

Introduction

Low Field Nuclear Magnetic Resonance (NMR) relaxometry techniques were developed in the laboratory to enhance and support comparable NMR logging tools that are currently used downhole. Low field NMR relaxometry has shown that discrimination of water and oil saturations in core and ore can be easily determined. In such cases, the NMR can detect the total water weight fraction and the total oil weight fraction, the viscosity of the oil, the amount of bound or mobile water, and the amount of mobile or bound oil[1-6].

Since these measurements are done in situ and the accuracy of the tools is at least as good as that of conventional extraction methods (i.e., Dean-Stark), NMR has emerged as a leading tool for heavy oil and bitumen characterization. Our research group has proved that many techniques that are routinely used in conventional oil could be extended to heavy oil and bitumen characterization[1-6]. In this paper, NMR is utilized for the determination of oil and water content of specific hydrocarbon streams. Of particular interest are the streams that contain emulsified fluids (water-in-oil or oil-in-water emulsions) which are currently very common in thermal production operations and are very difficult to handle. Test separators are currently used as the standard way of measuring the flow of thermally produced wells (CSS, SAGD, steam flooding). The test separators are inherently incapable of measuring emulsified flow. Other probe-type devices suffer from inaccuracies related to the presence of solids or gas, salinity, temperature, velocity, emulsion type, and range of cut.

Low field NMR emerges as the only currently proven alternative to discriminate accurately and precisely the amount of oil and water in such streams.

Methodology

The NMR spectra of a mixture of heavy oil or bitumen with water consist of two sets of T_2 relaxation peaks. The water peaks are at a range of 10 – 3,000 ms, while the oil/bitumen peaks are in the range of 0.2 – 10 ms at the specific temperature of 30° C. The degree of emulsification or separation of the phases, in addition to the temperature and the presence of additives, affects the ranges of these peaks. If the spectrum of a mixture is taken at a relatively low temperature, then the complete spectrum of the oil/bitumen may not be recovered.

If a known amount of a heavy oil (or bitumen) and water mixture is placed in the NMR, then the spectrum obtained can be deciphered in the spectra of the individual phases. The total obtained signal amplitude is compared to that of the same amount of a sample that only contains water (standard). If the amplitude of the standard is A_s and the amplitude of the water content of the unknown sample is A_w then the water fraction of the unknown sample is

$$W_w = A_w/AI_w \quad A_w = \sum A_j, j > 10ms$$

$$W_b = 1 - W_w$$

Where AI_w is the amplitude index of water standard of weight W_s

$$AI_w = A_s / W_s$$

This is the simplest way to calculate water and oil (or bitumen) content in any unknown sample of a known weight. If the whole spectrum of the liquids is obtained, then the oil (or bitumen) content can be obtained through the same equation as used for the water content, as long as the oil spectrum is corrected for its Amplitude Index (AI_{oil}) at a given temperature. The following equation applies:
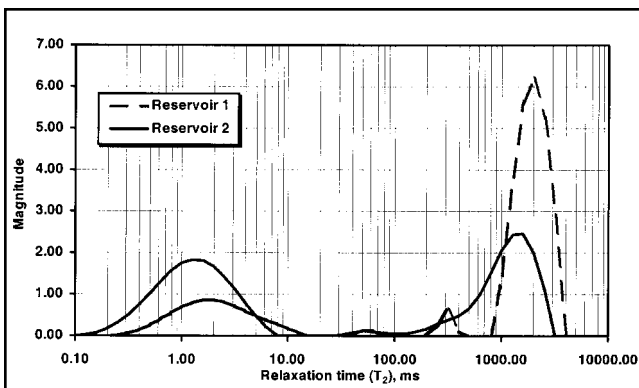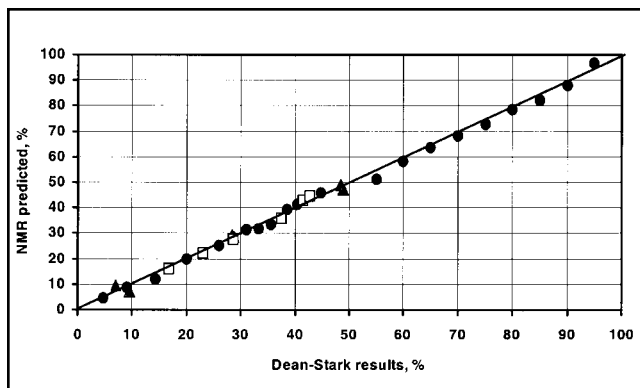
FIGURE 1: NMR spectra of stable emulsions.

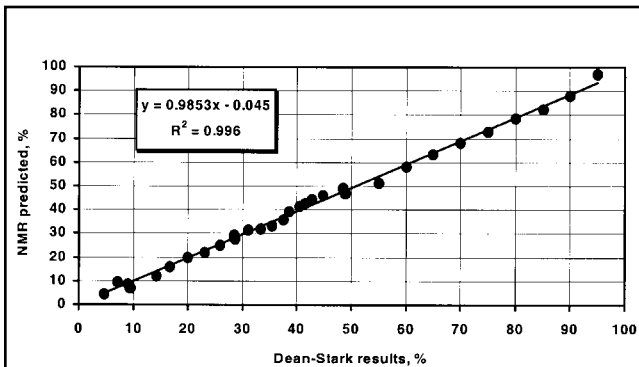**FIGURE 2: Water content prediction in water-oil mixtures.**

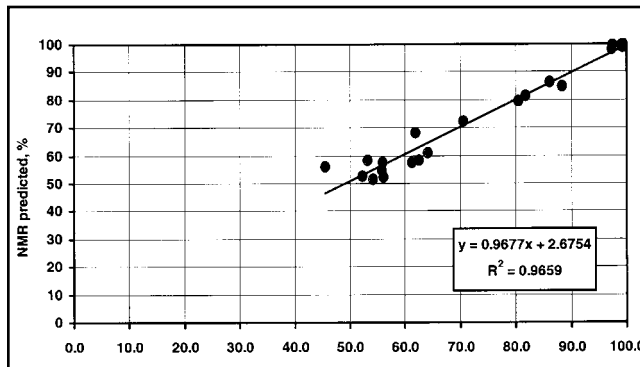**FIGURE 3: Correlation of water content prediction in water-oil mixtures (Reservoir 1).**

FIGURE 4: Correlation for water content prediction in water-oil mixtures (Reservoir 2).

$$W_{oil} = \left(A_{oil} / AI_{oil} \right) \quad A_{oil} = \sum A_j, \, j \leq 10ms$$

The second equation is valuable when the weight of the sample is not known and there is a suspicion of solids or gas present in the stream.

Demonstration of Concept

The capability of the low field NMR tool was demonstrated using samples from two different heavy oil reservoirs in Western Canada. Two different procedures were tested. For Reservoir 1, a variety of samples were prepared in the laboratory. The samples had water contents that covered the full range of the NMR. The samples were treated as "blinds." NMR testing was done, and the water content was calculated for all samples. The samples were then placed in the Dean Stark apparatus and the water content was determined. For Reservoir 2, samples were obtained from the wellhead, and they were brought into the laboratory for water content and oil content determination. First, NMR testing was done, followed by Dean-Stark measurements to verify the NMR results.

Figure 1 shows typical spectra from one sample from each reservoir. In both samples, it can be seen that the spectra is split in two parts, with a cut-off point of approximately 10 ms. The spectra are then manipulated as discussed above to provide the water and oil contents.

Figure 2 shows the comparison of NMR-predicted water content vs. Dean-Stark-measured water content for three different batches of samples from Reservoir 1. Although the batches were prepared at different times and were tested by different people, the results fall in the same line. In Figure 3, the same results are grouped and the trend-line is plotted. The correlation is excellent (0.996).

Figure 4 shows the comparison of the NMR-predicted data and the Dean-Stark measurement data for the samples of Reservoir 2. The correlation is also excellent (0.966). Figure 5 compares the results of Reservoir 1 and Reservoir 2. In Figure 6, the same results are grouped, and the common trend line is drawn. The overall correlation is 0.992, and it is deemed excellent.

Implementation

The experimental results presented above illustrate the potential of low field NMR for the accurate determination of oil and water content in heavy oil and bitumen streams. The fact that oil and water contents are estimated from the individual spectra components provides a set of measurements that are independent of
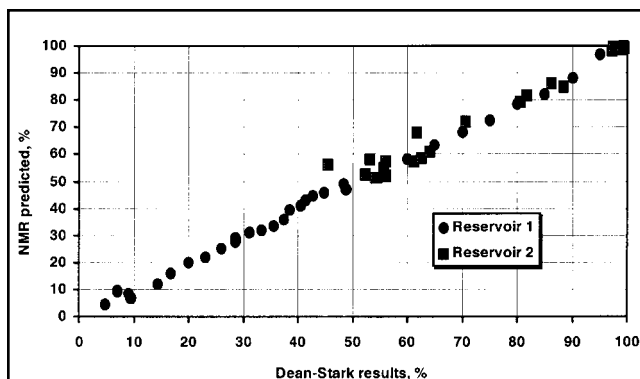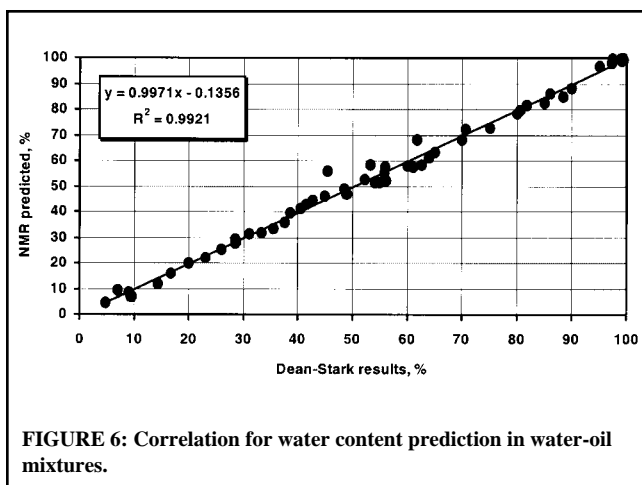

FIGURE 5: Correlation for water content prediction in water-oil mixtures.

**FIGURE 6: Correlation for water content prediction in water-oil mixtures.**

the presence of gas or solids. This is because both gas and solids (such as entrained sand) do not contribute to the measured spectra. This advantage of NMR technology sets it apart and ahead of any other type of measurement device in this area.

However, when these types of measurements are attempted in the presence of solids and/or gas, it is important to capture the complete spectrum of the oil. This is achieved by adjusting the temperature at which the measurements are taken. In general, an optimum temperature can be found in the range of 20 – 80° C that can be tailored for application in specific reservoirs.

Based on the successful laboratory results presented in this paper, a field prototype NMR-based system for direct measurement of water and oil fractions was designed and built. The system is designed to operate using slipstreams for the measurement of spectra of flowing streams on-line. Currently, the design allows for automatic sampling from a flowing stream, as is shown in Figure 7. Continuous flow sampling is also in the design stage. It is expected that this system will provide an accurate and fast measurement in the field.

Field Trials

Extensive field trials of the instrument have been performed over a four-month period. The instrument has been installed on a thermal recovery well pad and is located downstream of a two-phase test separator. The instrument has been mounted in a Class 1 Division 2 housing, and has been designed to withstand production streams up to 230° C and 4.14 MPa (600 psi).

FIGURE 7: Simplified schematic of the flow-through NMR system.
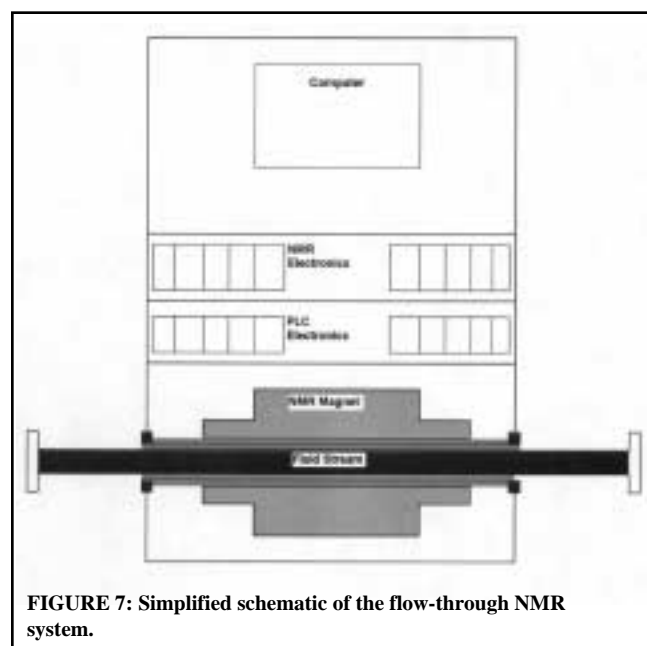
A trap and analyse system is being used, with the instrument located on a flow loop parallel to the main line. The flow is allowed go through the instrument loop, and then is blocked in with valves. A manual sample withdrawal system is also plumbed in, and samples have been withdrawn from this to validate the accuracy of the NMR measurement.

Although the system has proven able to handle fluid streams up to 230° C (maximum temperature encountered), analysis has only been performed at temperatures between 50° C and 100° C. This has been accomplished by allowing the sample to cool down in the instrument prior to analysis.

In summary the field trials have resulted in a trap and analyse system capable of the following:
- 230° C and 4.14 MPa (600 psi) maximum operating conditions.
- Analysis of samples at temperatures between 50° C and 100° C.
- The NMR technology has proven its ability to provide accurate in situ water and oil cuts.
- Accuracy of +/- 5% of conventional Dean Stark analysis (data not released by sponsor of field trial).
- Analysis may be performed in less than four minutes depending on in situ stream conditions.
- Sample cooling time is a limiting factor and varies depending on the initial temperature of the stream.
- NMR technology has been engineered to provide a stand alone automated analyser that can be tied into a computerized control system.

Based on the results of the field trials to date NMR technology offers the following advantages and disadvantages:

Advantages of NMR

- Accurate and repeatable measurements from 0 – 100% water cut.
- No need to recalibrate the instrument if the emulsion phase changes.
- NMR measurements are not affected by the following stream conditions:
  - Salinity
  - Density
  - Electrical conductivity
  - Emulsion consistency
  - Gas component
  - Vapour component
  - Solids component
- NMR can be placed off-line to replace centrifuge or Dean Stark laboratory analysis.
- NMR data can be manipulated to provide viscosity correlations for the produced oil.

Disadvantages of NMR

- Presently, analysis cannot be performed over 100° C.
- Cooling is required to bring samples down to "measurement temperatures."
- Cost of NMR is more expensive than most of the current range of inline measurement devices on the market.

Future Developments

- Minimize the temperature limitations of the NMR measurement algorithm. The upper measurement limit is mainly determined by the ability of the instrument to measure both the oil and water signal (at high temperatures the water signal moves out of the instruments detection limits.) Work is ongoing in this area.
- Decrease analysis time, software has been written that can decrease the analysis time to below two minutes. This software is being tested in the laboratory.

Conclusions

Low field NMR relaxometry was successful in accurately measuring water and oil contents in heavy oil and bitumen samples in the full range of compositions. The process was equally successful in both laboratory and field samples from two Western Canada reservoirs. The technology has been transferred for commercialization and field trials have been successfully carried out.

Acknowledgements

REFERENCES

1. MIROTCHNIK, K.D., ALLSOPP, K., KANTZAS, A., CURWEN, D., and BADRY, R., Low Field NMR Tool for Bitumen Sands Characterization: A New Approach; *SPE 71208 accepted, SPE Reservoir Evaluation & Eng., Vol. 4, No. 2, pp. 88 – 96, 2001.*
2. KANTZAS, A., MARENTETTE, D.F., ALLSOPP, K., and MIROTCHNIK, K., Quantification of Bitumen Using NMR; *US Provisional Application, 1999, Canadian Patent Docket No. 45074.3.*
3. MIROTCHNIK, K., KANTZAS, A., AIKMAN, M., and STAROSUD, A., A New Method for Group Analysis of Petroleum Fractions in Unconsolidated Porous Media; *Journal of Canadian Petroleum Technology, Vol. 40, (No. 7), pp. 38 – 44, 2001.*
4. MIROTCHNIK, K., and KANTZAS, A., Methods and Technology for the Characterization of the Pore Structure and Fluid Flow Properties of Soils in Reference to Contaminant Transport; *Journal of Canadian Petroleum Technology, Vol. 38, No. 11, pp. 41-47, 1999.*
5. MIROTCHNIK, K., KUBIKA, P., RANDALL, L., STAROSUD, A., ALLSOPP, K., and KANTZAS, A., Determination of Mud Invasion Characteristics of Sandstone Reservoirs Using a Combination of Advanced Core Analysis Techniques; *paper SCA 9815, presented at the International Symposium of the Society of Core Analysts, The Hague, The Netherlands, September 14 – 16, 1998.*
6. LATORRACA, G.A., DUNN, K.J., WEBBER, P.R., and CARLSON, R.M., Low Field NMR Determinations of the Properties of Heavy Oils and Water-in-Oil Emulsions; *Magnetic Resonance Imaging, Vol. 16, No. 5/6, pp. 659-662, 1998.*

Authors' Biographies

Kevin Allsopp is one of the co-founders of the TIPM Laboratory and currently serves as its business manager. Additionally, Kevin is the president of PERM Instruments, a company set up to commercialize Low Field NMR applications. Kevin has over ten years of R&D experience starting as a Research Technologist at Nova-Husky Research Corporation before moving into the business side of things at TIPM. Prior to this Kevin worked for ten years in the steel industry in the UK. Kevin has a diploma in chemical engineering technology from the Southern Alberta Institute of Technology and is currently working towards an MBA at the University of Calgary.

Ian Wright is a research engineer at the TIPM Laboratory. He has an M. Eng. degree in chemical engineering from the University of Calgary and a B.Sc. degree in chemical engineering from Queens University. Ian has been involved with the NMR commercialization project since its inception. He has been instrumental in designing the field applications and engineering a commercial NMR for oil/water measurements.

David Lastockin graduated from The Southern Alberta Institute of Technology with a chemical technologist diploma in 1989. He works for the TIPM Laboratory as a research technologist, primarily in the area of Low Field NMR applications. Dave was responsible for the implementation of the NMR field-testing program. Additionally, Dave has over six years of research experience.

Konstantin Mirotchnik is a research scientist in the Tomographic Imaging and Porous Media Laboratory. He received his Ph.D. in petrophysics, his M.Sc. in geophysics in the Moscow Oil and Gas University, Russia. In the past he has worked for the Central Geophysical Expedition (Moscow, Russia) as a senior geologist; with Numalog Ltd., (Israel, a subsidiary of Numar Co., USA), as a petrophysicist; with Givot Olam Ltd. (Israel) as a consultant in reservoir engineering; and with ONGC (India) as a consultant in geological applications of the Data Bank Control. His research interests include low field NMR applications for oil and gas reservoir studies, rock-fluid interactions, petrophysics, reservoir wettability characterization and the combined analysis of advanced core analysis results with petrophysical and DST data from fractured reservoirs.

Apostolos Kantzas is currently a professor at the University of Calgary and the director of the Tomographic Imaging and Porous Media Laboratory. He is also the Canada Research Chair in Energy and Imaging. Prior to this, he held a senior research engineer position in the Pipeline & Oil Technologies department at NOVA Research and Technology Corporation. He is involved in research related to problems of flow through porous media, enhanced oil recovery, soil remediation, reactor design, and tomographic imaging. He received a Dipl. Eng. in chemical engineering from the Aristotle University of Thessaloniki, Greece, in 1982, M.A.Sc. and Ph.D. degrees in chemical engineering from the University of Waterloo, Canada in 1985 and 1988, respectively. Dr. Kantzas is a member of APEGGA, SPE, the Petroleum Society, CSChE, AIChE, and SCA. He has authored or coauthored over 120 technical papers and over 100 technical reports.