

Aplicación de la resonancia magnética nuclear de bajo campo para estudiar la interacción entre un petróleo pesado colombiano y un solvente líquido

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Resumen

En este artículo se utilizó LF-NMR para estudiar la interacción entre un aceite pesado colombiano y un solvente líquido. Las pruebas de RMN se realizaron utilizando un relaxómetro Bruker Minispecmq7.5, operando a 7.5 MHz y otro equipo, operado a 1.75 Mz, para pruebas de reproducibilidad. La primera prueba implicó la determinación del coeficiente molecular de difusión para cuantificar el movimiento de moléculas de nafta y crudo pesado. Los resultados del coeficiente de difusión de la nafta en el petróleo pesado colombiano mostraron que la nafta se difunde rápidamente en el petróleo pesado en tiempos tempranos y se difunde a una velocidad más lenta con el aumento del tiempo. El coeficiente de difusión general promedio para el sistema se obtuvo después de 15 h con datos estables en un rango de $10^{-9} \text{ m}^2 / \text{s}$. La segunda prueba permitió estimar la densidad de diferentes mezclas mediante parámetros y correlaciones de RMN. En esta prueba se emplearon dos tipos de crudo pesado colombiano, cada uno diluido en nafta y tolueno y dos tipos de relaxómetro operados a 1.75 MHz y 7.5 MHz para evaluar la precisión de correlación a diferentes condiciones. Los resultados muestran una buena coincidencia al disminuir la densidad de la mezcla con un error porcentual promedio inferior al 5%.

Palabras clave: LF-NMR, densidad, viscosidad, difusión molecular.

Application of low field nuclear magnetic resonance to study interaction between a Colombian heavy oil and a liquid solvent

Abstract

In this paper LF-NMR was used to study the interaction between a Colombian heavy oil and liquid solvent. The NMR testings were done using a Bruker Minispecmq7.5 relaxometer, operating at 7.5 MHz and another equipment, operated at 1.75 Mz, for reproducibility tests. The first test involved the determination of diffusion molecular coefficient to quantify the motion of molecules of naphtha and heavy oil. The results of the diffusion coefficient of naphtha into Colombian heavy oil shown that naphtha diffuses into heavy oil fast in very early times and diffuses at a slower rate with increasing time. The average overall diffusion coefficient for the system was obtain after 15 h with a stable data in a range of was in a range of $10^{-9} \text{ m}^2/\text{s}$. The second test allowed to estimates density of different mixtures through NMR parameters and correlations. In this test were employed two kind of Colombian heavy oil, each one diluted in naphtha and toluene and two kind of relaxometer operated at 1.75 MHz and 7.5 MHz to evaluate the precision of correlation to different conditions. The results display good match by decreasing the density mixture with an average percentage error lower than 5%.

Keywords: LF-NMR, density, viscosity, molecular diffusion.

Introduction

In order to enhance heavy oil recovery, steam-based processes have been used in some Colombian fields (Maya et al, 2010), however, several negative technical issues are associated with this type of thermal based methods, such as large energy and water consumption, expensive water treatment, extensive heat losses and considerable greenhouse gas emissions (Zhongwei, Fanhua and Xialong, 2016). Alternatively, solvent-based methods are considered in many heavy oil reservoirs where steam is not suitable due to economic constraints and environmental concerns (Lin, Zeng, Ma, Gu, 2014), (Brayan, Nickel From, 2016).

The main driving mechanism in solvent based methods are reduction of viscosity and interfacial tension. These occur mainly in the mixing zone by the mass transfer mechanisms between the solvent and the crude (effective diffusion and mechanical dispersion), (Scheidegger, 1961). Effective Diffusion depends on molecular diffusion and porous medium properties). The molecular diffusion can be defined as the random motion of molecules from high concentration zones until low concentration zones, due to the concentration gradient between fluids in contact (Bachmat and Bear, 1964). The diffusion coefficient is the proportionality between flux and concentration gradient and it controls the mixture zone formation speed in solvent-based (Wen and Kantzas, 2005). For this reason it is considered the most important parameter to study solvent-based methods, because the knowledge of this concept allows to determine: the amount and rate of solvent injection, the portion of the reserves that have been affected by the solvent and the reduction of the viscosity and density of the crude, and the necessary time to allow

the solvent in contact with the crude oil and the production rate, (Perkins, 1963).

In this kind of process is very important to evaluate in the lab, the effect of solvent regarding oil properties and composition in order to estimate the effectiveness of the method and to avoid problems in the porous medium (Sandoval and Muñoz, 2019). This kind of experimental studies shows some problems because when the viscosity is too high, conventional measurements become less accurate and more difficult to obtain, raising the need for new rapid and non-destructive analytical tools and technologies. Low Field Nuclear Magnetic Resonance (LF-NMR) has been an attractive alternative to conventional measurements because it provides fast, unbiased, and non-destructive. Additionally, it allows to obtain the value of different properties with the same sample, at the same time.

In this paper, the measurement of diffusion coefficient was done with low-field nuclear magnetic resonance (LF-NMR), where it was observed that by diffusing the solvent and oil into each other; NMR properties as the amplitude and relaxation time of signals changed. These observations were later correlated to mass flux and concentration changes. Finally with these concentration changes and fick law the diffusion coefficient was calculated.

The NMR technique also was employed to measure the density of the mixtures of a Colombian heavy oil and naphtha, at different ratios and atmospheric pressure, to quantify the interaction between the solvent selected and Colombian heavy oil and to determine the reduction of this property. The density was estimated with a correlation that relates this property with relaxation time (T_2) and amplitude i relative hydrogen index (RHI).

Principles of low field NMR

As it is well known, when a sample, of NMR-sensitive nuclei (^1H , ^3He , ^{129}Xe ; ^{23}Na), is subjected to a uniform static magnetic field B_0 , a net macroscopic magnetization M of the sample appears in the same direction of B_0 . This magnetization is proportional, roughly speaking, to this polarizing field B_0 , to the density of nuclei within the sample and to the characteristic gyro-magnetic ratio (γ) of the nucleus being studied. In a typical one-pulse experiment, the sample is subjected to a short pulse (called excitation pulse) of a radiofrequency (RF) magnetic field B_1 , applied perpendicularly to B_0 and at the characteristic Larmor frequency (f). This frequency depends on the nucleus and of the static magnetic field according to the equation (1):

$$f = B_0\gamma \quad (1)$$

The effect of the excitation pulse is that the magnetization, M , is “tipped” or rotated from its initial direction (or from its thermal equilibrium state) by an angle α . This angle is called “flip angle”. It is proportional to field B_1 and to its duration, (τ), according to Equation (2):

$$\alpha = \gamma \tau B_1 \quad (2)$$

When the pulse is suspended, the absorbed energy by protons is released to the medium until reaching the equilibrium, process known as relaxation, in both transversal and longitudinal plane. During this process, the NMR signal-called also the Free Induction Decay (FID) - is received at the same frequency. This signal, which is proportional to the magnetization M (then to B_0) and to γ , is processed to be used for obtaining a “fingerprint” of the environment of the nucleus being studied.

Transverse relaxation time (T_2), is defined as the time taken by the transversal component of the magnetization vector to decay at 37% of its initial value (Bloch, 1946). This time is directly related to the viscosity of the fluids and the confining space i.e. when one fluid have high viscosity, its molecules are very close to each other, therefore they release faster the absorbed energy and their magnetization vector will take less time to align again with the external magnetic field. The average of the transverse relaxation time can be calculated with equation 3. (Afashi and Kantzas, 2006).

To determine the transversal relaxation time (T_2) sequences are used. The most employed sequences is CPMG (Carr-Purcell-Meiboom-Gill sequence), this type of spin echo pulse sequence consisting of a 90° radio frequency pulse followed by an echo train is induced by successive 180° pulses and it allows to obtain spin echo envelope amplitude, that is converted to spectrum after a numerical inversion with the inverse Laplace transform. The spectrum related the transversal relaxation time with the signal amplitude.

$$T_{2gm} = EXP \left[\frac{\sum AiLnT_{2i}}{\sum Ai} \right] \quad (3)$$

Where

T_{2i} = the value T_2 of each component in the spectrum
 A_i = Amplitude of each component at a constant time

The amplitude of the signal refers to the measurable hydrogen protons in the sample and it is proportional to the sample mass. The amplitude of the signal can be transformed to a mass value, using the concept of the amplitude index (AI), which corresponds to the slope of the straight line obtained in the graph of the amplitude measured in the NMR equipment and the mass of the analyzed fluid (protons), Equation 4.

$$AI = \left[\frac{A}{m} \right] \quad (4)$$

Where

A = total amplitude of sample
 m = sample mass

Considering that not all NMR equipment is operated at the same signal gain, so the value of oil amplitude index could vary from machine to machine. In order to normalize the measured amplitude index, the term relative hydrogen index is defined as the relation between amplitude indexes of the analyzed sample (*AI_{sample}*) and the water (*AI_{water}*) at the same acquisition conditions, as it is shown in Equation 5.

$$RHI = \left[\frac{AI_{sample}}{AI_{water}} \right] \quad (5)$$

Determination of properties with NMR

Molecular diffusion

The molecular diffusion is the random flow of components due to the concentration difference between two fluids (Salama and Kantzas, 2005). This process causes that the initially sharp interface among the two fluids will become a diffused mixed zone gradient from one pure fluid to the other. The molecular diffusion is described by the Fick's law, as shown in Equation (6), that allows to analyze the concentration change with respect to the distance and time with a proportionality constant, it is known as molecular diffusion coefficient (Bird, Stewart and Lightfoot, 2002).

$$\frac{\partial C}{\partial t} = D_o \frac{\partial^2 C}{\partial x^2} \quad (6)$$

The initial condition for $t > 0$ is

$$C = C_s \text{ for } x < 0$$

$$C = C_o \text{ for } x > 0$$

The boundary condition for $t > 0$ is

$$C = C_s \text{ at } x = -\infty$$

$$C = C_o \text{ at } x = \infty$$

Where,

C_s = Solvent concentration

C_o = oil concentration

t = time

x = diffusion depth

For free diffusion, the coordinate is assumed to be positive in the downward direction and is measured from the vertical position in the vial. At the interface between solvent and oil, x equals to zero. (See **Figure 1**) (Wen, Bryan and Kantzas, 2003).

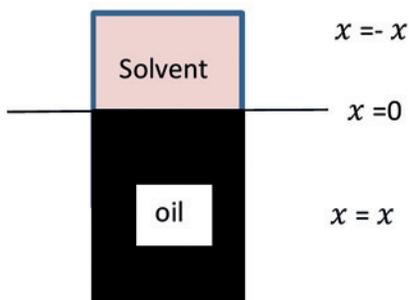


Figure 1. Diffusion process and definition of initial boundary conditions.

Using equation 6 and boundary conditions, it is possible to obtain Equation 7. That allows to determine the change of concentration along the x -axis with a fixed solvent/oil interface, at a fixed value of the diffusion coefficient D_o at a specified time of one of the components in the system in terms of the distance and time. This equation assumes that the diffusion is only in one direction, in an infinity medium, where the density is constant.

$$C = C_i \left[\operatorname{erfc} \left(\frac{x}{2 * \sqrt{D_o * t}} \right) \right] \quad (7)$$

Where

C = concentration of diffusing substance, mass fraction

C_i = initial concentration of diffusing substance at time zero, mass fraction

x = diffusion depth, cm

D_o = Diffusion coefficient cm^2/s

Procedure to determine diffusion coefficient

As explained above, based on the characteristics of low-field NMR it is possible to determine the fraction of liquids present in a mixture if there are significant differences in their relaxation times. Because the heavy oil and liquid solvent have a wide difference between its viscosities, and therefore in their relaxation times, it is possible to identify

each fluid and the mixture of both in the spectrum and with this information determine the amount of solvent that would have diffused into the heavy oil in a certain time.

To achieve the main objective of this work, naphtha and a Colombian heavy oil were employed (see **Table 1**). Each one pure fluid was inserted into the low field NMR equipment in 5 cm diameter tubes at a temperature of 40 ° C with the NMR parameters shown in **Table 2**.

Fluid	Viscosity 30 °C (cp)	Density 30°C (g/cm ³)
Naphtha	0,10	0,681
Heavy oil	36119.40	1.0004

Table 1. Properties of fluids.

These NMR parameters were carefully selected as a poor choice could lead to an overestimation or an underestimation of the diffusion coefficient. For this study, the selection of parameters began with an overview of previous research (Barbosa et al, 2017) (Niu, Kantzas, and Bryan, 2008). These values were subsequently sensitized to choose the parameters that would allow the identification

of solvent and heavy oil signals. One of the most critical parameters for the determination of this property was the time of the echoes, since with a very long time it would not be possible to determine the oil signal. For this case, due to the difference in viscosities between the fluids, it was decided to work with the lowest echo time allowed by the application, that is, 0.01 ms.

NMR parameter	Value
Echoes time (ms)	0.01
Number of echoes	8000
Recycle delay(s)	18
Numbers of scans	32

Table 2. NMR parameters for diffusion experiment.

According to the nature of every fluid, the signals were easily discriminated. The resulting spectra show that the naphtha signal was located in relaxation times greater than 1000 ms and the heavy oil in times less than 100 ms as can be seen in Figure 2.

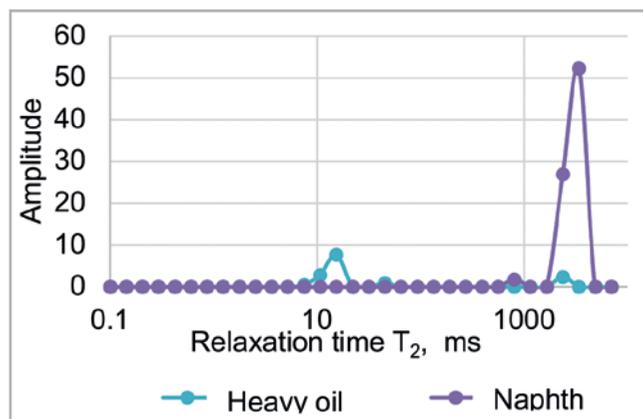


Figure 2. NMR spectrum oil 1 and naphtha. Source: Authors.

After NMR properties of each pure fluid were estimated, mixtures with different amount of naphtha were prepared and taken to the NMR equipment to analyze the effect of naphtha concentration on properties of mixture signal.

It was possible to conclude, that the mixture signal shifts towards longer relaxation time by increasing the naphtha concentration, i.e the relaxation time increase with the solvent concentration, (see **Figure 3**)

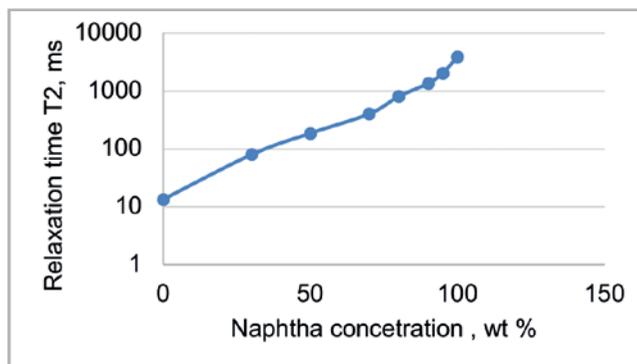


Figure 3. Semi-log plot of average relaxation time vs solvent concentration- relaxometer at 7.5 MHz.

According to the information above, the cut-off relaxation time (T_{2c}) was determined, which is defined as the maximum relaxation time for each mixture (naphtha and oil). This time divides the spectrum into two parts, allowing to determine the amount of diffused solvent in oil. Then, the signals with

less relaxation time than T_{2c} correspond to oil and solvent. The signals with relaxation time greater than T_{2c} correspond to the pure solvent that has not been diffused, as can be seen in **Figure 4** for Colombian heavy oil and naphtha.

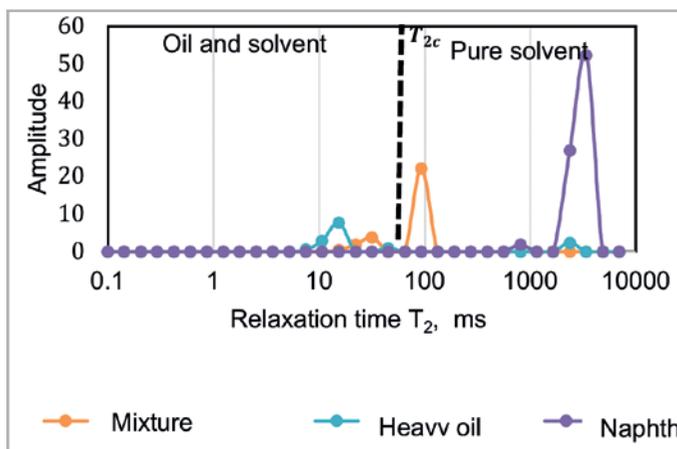


Figure 4. The cut-off relaxation time for a mixture with oil 1 and 16% naphtha.

naphtha/oil (%p/p)	t_{2c} (ms)
16	133
30	201
50	396
70	779

Table 3. Cut-off relaxation time for different mixture of naphtha and oil 1.

Afterward to estimate the Cut-off relaxation time, the diffusion process was started, using NMR tubes of 10 cm high and 5 cm in diameter, employing the equipment mini spec 7.5 at a constant temperature of 40 °C and atmospheric pressure. First, the heavy oil sample was placed at the bottom of tube with a height of 2 cm, then the naphtha was carefully placed on the top.

Afterward placing the fluids into the tubes, NMR measurements were taken frequently, (see **Figure 5**), observing that for every time were obtained spectrums with different relaxation times and amplitudes of pure naphtha

signals, because the amount of this solvent decrease in the time, since it was spreading in the oil. To determine the amount of pure naphtha at each time, it was necessary to estimate the amplitude index of pure naphtha ($AI_{naphtha}$). In this way, with the amplitude of the signal corresponding to each time, the amplitude index of pure naphtha and using Equation 4, the amount of pure solvent that has not diffused into heavy oil was calculated. Subsequently, this value was subtracted from the initial mass of solvent and the mass of the naphtha diffused in the heavy oil was obtained. **Table 4** shows the concentration of naphtha diffused in the heavy oil for different times.

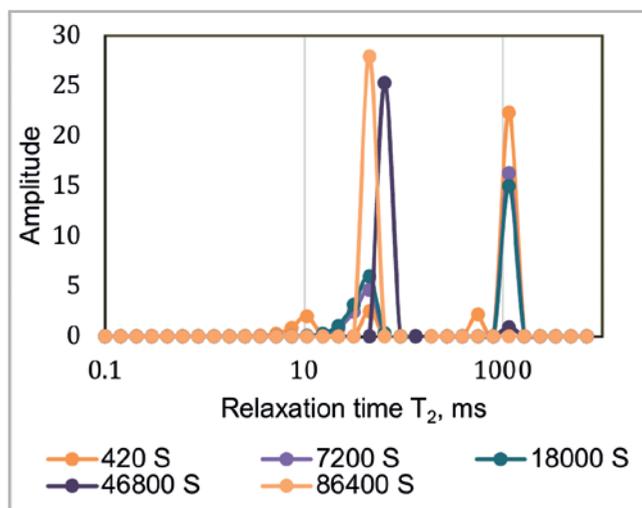


Figure 5. Change of NMR spectrum as naphtha diffuses in oil heavy oil.

Times(s)	Naphtha/oil Concentration (%p/p)	Diffusion coefficient (m ² /s)
420	5.521	1.30E-07
3600	6.936	1.76E-07
7200	8.572	9.41E-08
10800	8.801	6.36E-08
18000	8.857	3.65E-08
46800	13.33	1.90E-08
54000	12.863	1.50E-08
61200	13.473	1.34E-08
72000	14.195	1.16E-08
86400	15.878	1.22E-08

Table 4. Results of diffusion coefficient of naphtha into oil 1 at different times.

With these values of diffused solvent concentration in time and Equation 7, taking the value of the distance constant and equal to 1 cm, the diffusion coefficient was calculated as a function of time. It should be mentioned that measurements were taken in a static state, and no mechanical stirring was involved.

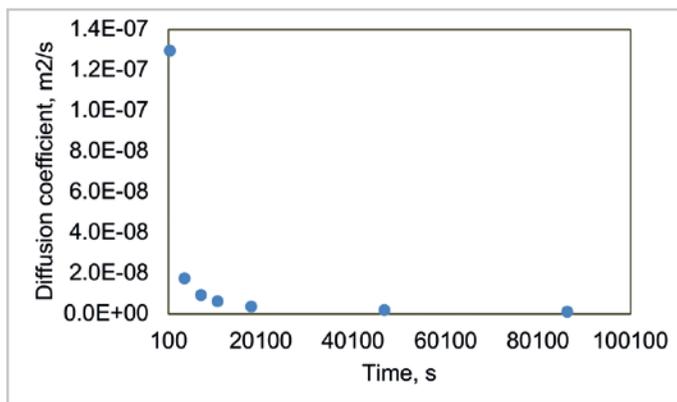


Figure 6. Change of NMR spectrum as naphtha diffuses in oil 1.

The results of the diffusion coefficient of naphtha into Colombian heavy oil are observed in Figure 6 and Table 4. It is shown that naphtha diffuses into heavy oil fast in very early times and diffuses at a slower rate with increasing time. The diffusion coefficient is in range of 10⁻⁷ m²/s at the early times. In the first hour during the diffusion process, the diffusion coefficient decreases slowly and becomes

stable in the range of 10⁻⁹ m²/s. To obtain the average overall diffusion coefficient for the system, stable data of diffusion coefficients were selected, after 15 h. The final results in Table 4, compared to literature data, shows the overall diffusion coefficients obtained from NMR are in a range similar to those investigations previously observed. (Wen, Bryan and Kantzas, 2003), (Wen and Kantzas, 2005).

Solvent and heavy oil/bitumen mixture

To describe the behavior of each one the mixture of Colombian heavy oil and naphtha, several dilutions were prepared at different proportions (see Table 6), employing a vortex shaker for almost 6 hours.

The viscosities of these mixtures were estimated at 30 °C using Brookfield LVDV-III Ultra-model viscometer. The

densities were measured using Anton Parr DMA 4100 densimeter at the same temperature conditions.

The prepared samples were taken to the NMR equipment relaxometer (1.75 MHz of frequency) and processed with the acquisition parameters shown in **Table 5**. These parameters were obtain from previous research using similar fluids to determine viscosity or density (Barbosa et al, 2017) (Wen and Kantzas, 2005).

NMR parameter	Value
Echoes time (ms)	0.2
Number of echoes	5000
Recycle delay(s)	12
Numbers of scans	32

Table 5. NMR parameters for mixtures heavy oil and solvent.

The NMR spectra of the mixtures (**Figure 7 and Table 6**) shows that increasing the solvent concentration, the relaxation time increase, since it is inversely related to the

viscosity. Also, it can be seen that the amplitude increases due to the increase in the amount of hydrogens by the addition of naphtha.

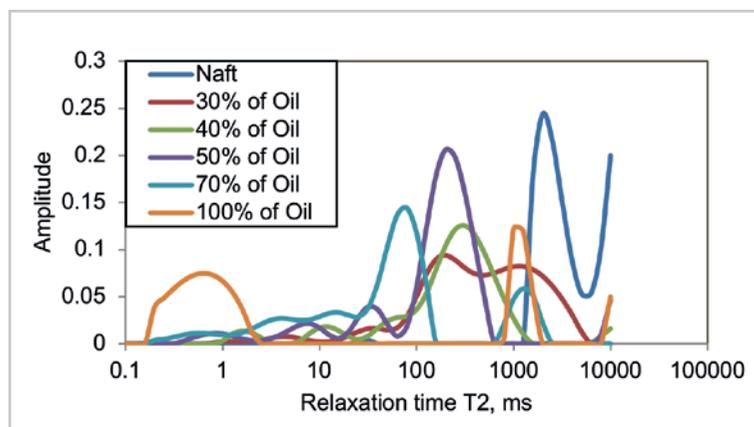


Figure 7. NMR spectrum of each mixture.

The results indicate that increasing the solvent concentration, the viscosity and density values of the mixture decrease, as it is shown in Table 6. In addition, it was possible to identify

a change in the properties of amplitude and average relaxation time (T_{2gm}) obtained from NMR.

% wt solvent	Density (g/cm ³)	Viscosity (Cp)
100	0.6581	0.10
70	0.7319	2.82
60	0.7686	8.41
50	0.8117	27.83
30	0.8643	250.23
0	1.0004	36119.40

Table 6. Viscosity, density and NMR properties of the mixtures.

Figure 8 shows that there is a semi logarithmic relationship between the concentration of naphtha and T_{2gm} . On the other hand in **Figure 9**, it can be analyzed that there is a linear relationship between the concentration of naphtha and RHI.

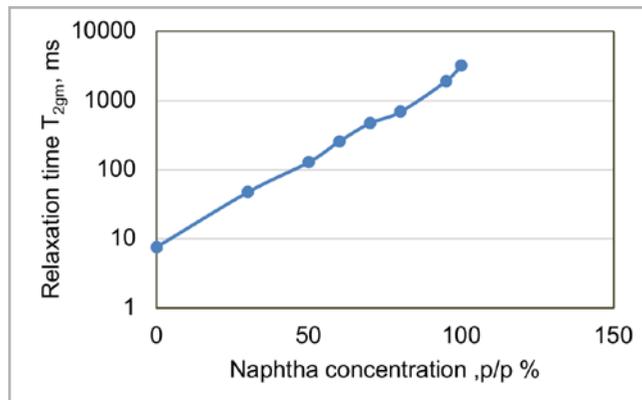


Figure 8. Semi-log plot of Average relaxation time vs solvent concentration, relaxometer at 1.75 MHz.

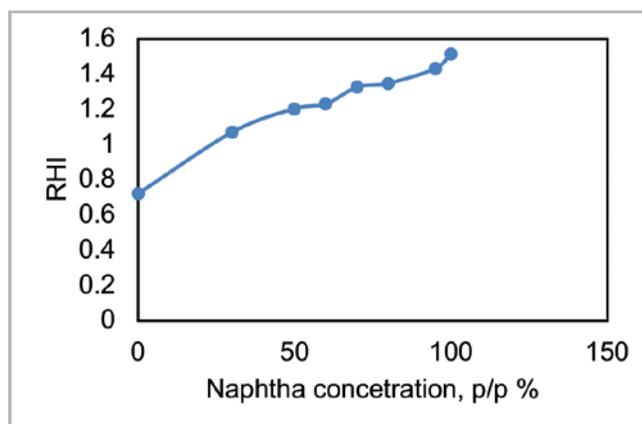


Figure 9. Relative hydrogen index vs. solvent concentration-relaxometer at 1.75 MHz.

Information included in **Figures 8** and **9** allow the definition of a relationship of the concentration of the solvent with the NMR properties, as presented in equation (8):

$$Cs = b' * \log(T_{2gm}) + c' * RHI + a' \tag{8}$$

Slope values b' , c' and intercepts a' were estimated after doing a multivariate regression with the Excel tool and the results are shown in **Table 7**.

Constants	Values
a'	-36.54
b'	37.62
c'	5.00

Table 7. Values of slope and intercepts- relaxometer at 1.75 MHz.

Density determination by NMR model

The data of **Table 6** indicate that the density of the mixtures decreases linearly as the solvent content increases. In addition, the results show that the inverse of the geometric

mean of T_2 (T_{2gm}) and the relative hydrogen index (RHI) values change, as the solvent concentration increases, therefore the change in the density of the mixtures affects the NMR properties, monotonically. (See **Figure 10**.)

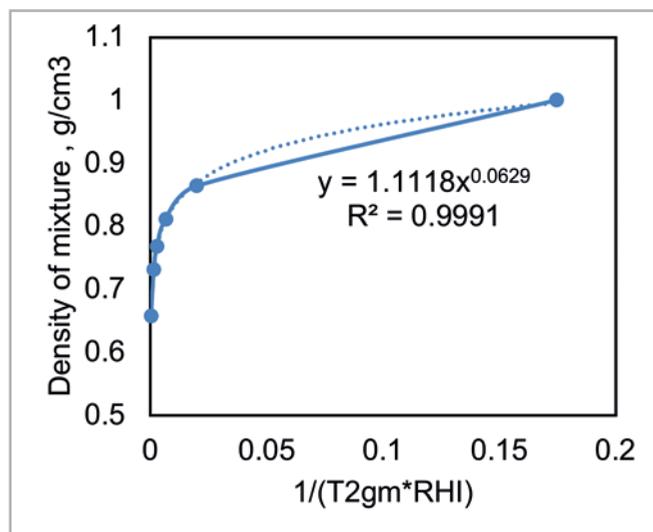


Figure 10. Correlation of densities of the mixtures of Colombia heavy oil with naphtha with the NMR parameter.

The NMR density model is expressed as equation 9, where the NMR properties (T_{2gm} and RHI) are related with mixture density α and β and are fitting constants (1.1118 and 0.0629 respectively). This model fits with those found in the

literature (Wen and Kantzas, 2005) and allows to estimate the mixture density with NMR for different proportions of naphtha and the Colombian heavy oil.

The comparison between measured density and the calculated density for the mixtures of Colombian heavy oil with naphtha was plotted in **Figure 11** and shown **Table 8**. The results show that the density values obtained with the NMR model had a good adjustment with respect to the one estimated in the laboratory, with an average percentage error equal to 1.8% and a correlation coefficient close to 1, which indicates a positive correlation between the viscosities obtained. These results show that NMR can be a useful tool to predict density changes as the heavy oil mix with liquid solvent.

$$\rho_{mezcla} = \frac{\alpha}{(T_{2gm} RHI)^\beta} \quad (9)$$

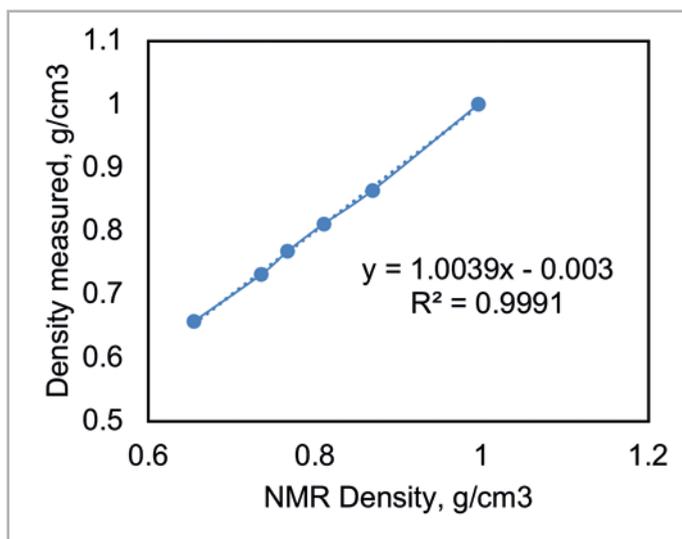


Figure 11. Comparison between NMR densities and measured densities for a mixture of naphtha and Colombian heavy oil- Relaxometer at 1.75 MHz.

Density (g/cm ³)	NMR Density (g/cm ³)	Relative error(error)
0.658	0.656	0.395
0.732	0.775	5.895
0.769	0.779	1.311
0.812	0.814	0.334
0.864	0.874	1.087
1.000	1.005	0.456
	Average	1.80

Table 8. Comparison between NMR densities and measured densities for a mixture of naphtha and Colombian heavy oil.

To show the precision of the estimated constants to use density correlation, a sample of other Colombian crude is diluted in different proportions with toluene. These mixtures were taken to the equipment with a frequency of 7.5 MHz and the results are presented in **Figure 12** and **Table 9**.

%wt solvent	T2gm	RHI	Conventional density measured (g/cm ³)	NMR Density (g/cm ³)	Relative error (%)
0	4.011	0.569	0.972	1.056	8.643
2	4.618	0.630	0.968	1.040	7.331
4	5.888	0.705	0.968	1.017	5.037
6	6.302	0.781	0.964	1.006	4.291
8	7.031	0.865	0.962	0.992	3.09
10	8.913	0.943	0.957	0.972	1.574
12	10.513	0.985	0.955	0.960	0.530
Average error (%)					4.35

Table 9. Comparison between NMR densities and measured densities for a mixture of toluene and Colombian heavy oil.

The results obtained indicate that the largest errors reported are related to the samples with higher density. However, increasing the amount of toluene the adjustment improves, reaching a relative error percentage lower than

5%. **Figure 12** presents a good correlation coefficient of 0.99 among the conventional measurement of density vs NMR density for mixtures with low density.

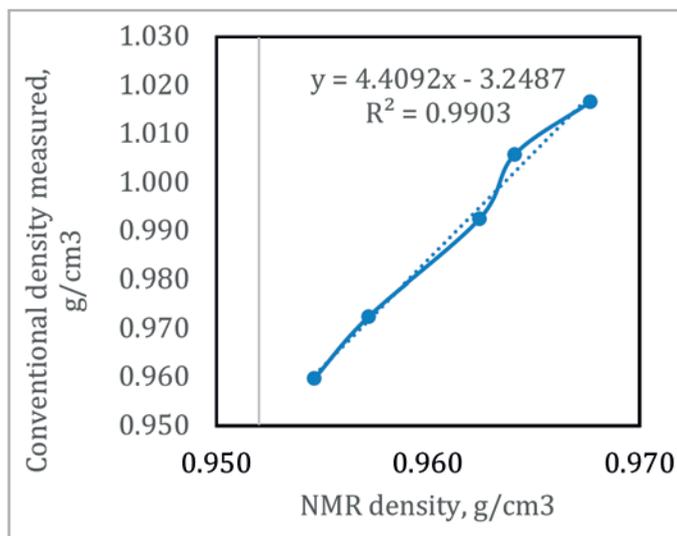


Figure 12. Comparison between NMR densities and measured densities for a mixture of toluene and Colombian heavy oil-Relaxometer at 7.5 MHz.

Conclusions

The NMR show to be a useful tool to study solvent-heavy oil interaction because is very fast, non- intrusive technique. It allows to obtain the value of different properties with the same sample at the same time.

The results of the diffusion coefficient show that naphtha diffuses into heavy oil fast in very early times and diffuses at a slower rate with increasing time. The diffusion coefficient is in range of 10^{-7} m²/s at the early times. In the first hour during the diffusion process, the diffusion coefficient decreases slowly and becomes stable in the range of 10^{-9} m²/s after 15 hours.

The NMR spectra of the mixture shown that increasing the solvent concentration, the relaxation time increase, since it is inversely related to the viscosity. Also, it was observed that the amplitude increases due to the increase in the amount of hydrogen by the addition of naphtha.

The correlation to determine mixture density show a good match with an average relative percentage lower than 5%. Even when the type of mixture changed and the frequency of the magnetic field shifted from 1.75 MHz to 7.5 MHz.

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