A model of capillary equilibrium for the centrifuge technique

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This paper addresses the problem of modeling transient production curves during a centrifuge experiment. Modern accurate measurement techniques used to monitor fluid saturation versus time while spinning indicate that capillary equilibrium is difficult to define and that a stability criterion for changing the centrifuge speed is needed. Such a criterion is very often set empirically, based on individual experience. This approach can lead to errors in the capillary pressure curves that are not detected by capillary pressure interpretation algorithms.

We propose a mathematical model using two exponential functions to fit the measured production curves. The bi-exponential model is necessary because of the existence of two typical time scales, that are different by a factor of ten. They are interpreted as the signature of viscous and capillary-controlled flow. For constantly accelerating centrifuge experiments, a model using two strechted exponential functions is proposed.

The bi-exponential model can be used to reduce experimental time while providing a rigorous criterion to change the speed of rotation of the centrifuge, according to the user's accuracy target. The 4 parameters of the model are calculated while measuring and the stability of the extrapolated equilibrium saturation is the criterion for changing the centrifuge speed. The proposed methodology is demonstrated using various examples of drainage and forced imbibition experiments performed using an automated centrifuge. We show that, compared to an experiment where equilibrium is reached within measurement accuracy, the duration can be reduced by a factor of two with negligible loss of accuracy.

INTRODUCTION

The centrifuge technique is one of the most important tools in special core analysis studies. It is used at various degrees of automatization for measuring capillary pressure curves, wettability indices and more recently relative permeability curves. Using very simple equipment, it can also be used to desaturate samples either in drainage to estimate irreducible water saturation or in forced imbibition to estimate residual oil saturation. There are various technical advantages related to the centrifuge technique: (i) the absence of semi-permeable filters (ii) the ability of analyzing 4 to 6 samples using a single machine (iii) a short experimental time compared to other methods. Among the drawbacks is that the temperature is limited to about 80°C in most experimental set-ups and that a confining pressure on the sample to reproduce field stress effect is technically very difficult to achieve. Also, the existence of a non uniform saturation profile requires that data must be interpreted using an appropriate method (see Forbes, 1997, for a review), and the

spontaneous imbibition curve cannot be measured. There are various attempts to overcome the above limitations and to extend the centrifuge method to the measurement of the full imbibition curve (Fleury et al., 1999) and therefore, there is still a large potential for improvement.

The purpose of this paper is to address the problem of capillary equilibrium which influences the duration of the centrifuge experiment and the accuracy of the capillary pressure curves. Despite the widespread use of the centrifuge technique in the oil industry, there is no clear methodology to set the duration of a speed step or define when capillary equilibrium is reached. The problem of capillary equilibrium has been addressed by King (1990) and Hirasaki (1993) and it is expected that this work will further contribute to setting quality standard among the users of the centrifuge technique. As will be seen below and from our experience, our observation shows that capillary equilibrium is seldom reached within a reasonable time in a centrifuge experiment and that the notion of capillary equilibrium is very ambiguous. Note that capillary equilibrium is also difficult to define in other experiments such as the porous plate or micropore membrane experiment. For these experiments, Fleury et al. (1997) proposed to fit transient saturation curves between (pseudo) equilibrium points using a single exponential curve, based on experimental and theoretical considerations. There is a similar approach in this paper and we propose the use of two exponentials to fit transient saturation curves.

We first present the model in two different experimental situations (fast and slow acceleration). The relevance of the model is illustrated using both experimental and numerical data. Then, a method is proposed to optimize the centrifuge experiment in terms of duration.

DESCRIPTION OF THE MODEL

The model has two formulations depending on the experimental conditions. When the speed of rotation of the centrifuge is increased rapidly (of the order of 1000 rpm/min), this mode is called fast acceleration and the transient saturation curves are described using 4 parameters. When the centrifuge speed is changed gradually (for example 5 rpm/min), this mode is called slow acceleration and an extended formulation with 6 parameters is used. The latter mode is not standard and is mostly dedicated to the determination of relative permeability curves. It will not be discussed in great details.

Fast acceleration

We consider here a multi-speed experiment where the average saturation of the sample is measured as a function of time with a constant speed of rotation. Several analytical expressions based on combinations of exponential functions as well as other functions (erf, power laws) were tested. Finally, we chose the following form:

$$S(t) = S_{ini} + \Delta S_{eq} \left[1 - \left(w \times \exp(-\frac{t}{T_1}) + (1 - w) \times \exp(-\frac{t}{T_2}) \right) \right]$$
 Eq. 1

Using experimental data, four different parameters must be determined:

- $\blacktriangleright \Delta S_{eq}$ is the saturation variation during the speed step considered.
- \succ T₁ and T₂ are characteristic times of the exponential functions.
- \blacktriangleright w represents the weight affected to the first exponential.

As will be seen below, it appears that exponential functions are well adapted to capillary pressure experiments (porous plate, centrifuge) where the driving force (the imposed pressure difference between oil and water) is gradually reduced by the response of the sample (a change of saturation as given by the capillary pressure curve). When considering gravity drainage problems, it can be shown (Pavonne, 1989) that the saturation as a function of time can be calculated analytically as a series of exponential functions, strongly suggesting the model of Eq. 1. King et al. (1990) also chose exponential functions but selected a stretched exponential to represent the deviation from a single exponential (therefore assuming a broad spectrum of decay rates). Hirasaki (1993) considered a expression essentially based on a power law function where the exponent is linked to the Corey exponent modeling the relative permeability curve of the displaced fluid.



Figure 1: Experimental data from an air-water 1^{st} drainage modeled by three models involving one, two and three exponential functions. The latter does not improve the fit, and the former is clearly insufficient. Sample: Vosges sandstone, K=680 mD.

When considering experimental production curves (Figure 1, taken from an airwater first drainage experiment, see Fleury et al. 1998, Figure 5), two regions can be clearly distinguished: at short times, there is a large variation of saturation, suggesting that dynamic effects are preponderant; for long times, capillary forces become of the same order of magnitude as viscous forces, leading to capillary equilibrium. These two mechanisms have two characteristic times, which correspond to the bi-exponential form. We checked on this example that two exponential functions are sufficient to properly model experimental data (Figure 1 and Table 1). The single exponential model is clearly insufficient while a three exponential model does not bring significant improvement compared to the bi form. The two characteristic times T_1 and T_2 are different by a factor of ten (Table 1).

	Exponential n° 1		Exponential n° 2		Exponential n° 3		
Parameters	$T_1(h)$	W	T ₂ (h)	W	T ₃ (h)	w'	Standard error
Uni-exponential	0.890	1.000					1.441E-02
Bi-exponential	0.120	0.654	3.894	0.346			2.46E-04
Tri-exponential	0.000	0.283	0.208	0.382	4.005	0.335	2.115E-04

Table 1: Comparison of different models using one, two or three exponential functions.

The model is further validated on two oil-water forced imbibition and 2nd drainage experiments performed on a carbonate sample of intermediate wettability (USBM wettability index of -0.15). These experiments were performed using a low speed centrifuge equipped with a capacitance based level detector, as described in Fleury et al. (1998). The experimental conditions are summarized in Table 2. The 4 parameters of the model in equation 1 were calculated using standard optimization routines with initial values $T_1=1$ hr, $T_2=10$ hr, w=0.5 and ΔS_{eq} the final saturation change obtained at a given step. For all the steps and for both experiments, the model can fit the experimental data with a very good accuracy. The two characteristic times of the exponential functions are also clearly distinguished: for each step, there is a rapid change of saturation after the change of speed and the order of magnitude of T₁ is 0.5 hr. T₂ values are much larger (about 10 hr) and more uniform over the entire experiment. For the second drainage experiment (Figure 3), the behavior is similar. As an exception, step 1 is characterized by a much shorter T_2 which may have been influenced by a spontaneous drainage process occurring before the start of the experiment (not shown). For both drainage and imbibition, T_1 and T_2 are different by more than a factor of ten.

Sample type	Carbonate			
Porosity (% of pore volume)	24.6			
Water Permeability	107 mD			
Length	5.8 cm			
Diametre	3.95 cm			
Max radius in drainage	18 cm			
Max radius in imbibition	25 cm			
Speed sequence (forced imb.)	200-250-300-400-500-600-900-1400-2000- 2700			
Speed sequence (2 nd drainage)	200-300-400-500-600-1000-1500-2500-2800			
Spontaneous drainage	5%			
Spontaneous imbibition	2%			

Table 2 : experimental conditions for the forced imbibition and 2nd drainage shown in Figure 2 and Figure 3.



Figure 2: forced imbibition experiment on a carbonate sample of intermediate wettability. Both the experimental data and the fit are plotted. For the step 5 shown in detail in the lower right corner, the circles correspond to the experimental data and the line to the fit. For each step, the parameters of the fit are indicated (T_1 and T_2 in hr). S_{fin} is the saturation measured at the end of the step, which should be compared to the extrapolated saturation S_{eq} . Sample : carbonate, K=105 mD, wettability index -0.15.

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Figure 3 : second drainage experiment on a carbonate sample of intermediate wettability. Both the experimental data and the fit are plotted. For the step 4 shown in detail in the upper right corner, the circles correspond to the experimental data and the line to the fit. For each step, the parameters of the fit are indicated (T_1 and T_2 in hr). S_{fin} is the saturation measured at the end of the step, which should be compared to the extrapolated saturation S_{eq} . Same sample as in figure 2.

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Slow acceleration

The slow acceleration mode is of interest when extracting relative permeability curves from transient saturation data (see Ruth, 1997 for the latest advances). A slow acceleration amplifies the difference between two sets of relative permeability curves and therefore provides more accurate estimation. The bi-exponential form can be extended to the case of slow acceleration using two stretched exponentials as follows:

$$S(t) = S_{ini} + \Delta S_{eq} \left[1 - \left(w \times \exp\left[-\left(t / T_1 \right)^{n_1} \right] + (1 - w) \times \exp\left[-\left(t / T_2 \right)^{n_2} \right] \right]$$
Eq. 2

In this case, we used numerical simulations to validate the above formulation. The example shown in Figure 4 is the case of an acceleration of 5 rpm/min, which is the lowest value possible in our experimental set-up. The maximum speed of rotation was set to 2000 rpm. The numerical data and the model are in good agreement.



Figure 4: modeling of slow acceleration experiment. Left panel: example where the speed of rotation is increased from 0 up to 2000 rpm at a rate of 5 rpm/min. Right panel: evolution of the exponents n_1 and n_2 (Eq. 2) with the value of acceleration

The exponents n_1 and n_2 are linked to the value of acceleration, as indicated by Figure 4 (right panel). When acceleration increases and tends to a fast acceleration mode, both exponents tend to one. For very small acceleration (<50 rpm/min), the fluctuations of the calculated values indicate that n_1 and n_2 may not be independent. Further work is needed to extract a relationship between n_1 and n_2 for a wide range of experimental conditions.

METHODOLOGY FOR OPTIMIZING THE CENTRIFUGE EXPERIMENT

Reaching 'true' equilibrium may take large amount of time because T_2 are large and influence significantly the shape of transient curves. In practice, before reaching the precision of the measurements, one will always detect a change of saturation. With accurate measurement systems such as the one used in our experiment (the sensitivity is

better than 0.5% of the pore volume), the question of the speed change criterion arises. King et al. (1990) also considered this issue and suggested a duration of 24 hr as a guideline for typical samples (here Berea). IFP's standard is a duration of 16 hr for a 100 mD sample. This duration is then modified in real time to take into account the absolute and (unknown) relative permeability of the sample analyzed. Therefore, a typical duration for a centrifuge experiment (drainage or forced imbibition) lies between 7 and 10 days depending on individual experience and measurement systems, assuming that 8 to 10 points are necessary to properly determine a capillary pressure curve (SCA guidelines). However, when low permeability samples are considered (1-10 mD), the duration will theoretically not be practical (of the order of one month) or economically irrelevant. For these samples, the extrapolation method for determining equilibrium saturation is critical. We propose here a methodology to shorten the experimental time while keeping sufficient accuracy.

Description of the method

The basic idea is to extrapolate the equilibrium saturation with the least measurement time at each step, therefore searching for the best compromise between duration and accuracy. The extrapolation of the equilibrium saturation is essentially driven by T_2 , which can be determined quite rapidly.



Figure 5: Production curves measured during two experiments on the same sample. In the short experiment, the duration of the speed steps was shortened and the equilibrium saturation extrapolated. There is a considerable reduction in duration. Sample : sandstone, Kw=653 mD, Φ =17.1 %. Fluids : brine, dodecane.

We performed a specific experiment to test whether the rotation speed can be changed far from equilibrium. An experiment was first performed using very long steps (Figure 5) to obtain a very good estimation of the equilibrium saturation. Then, the experiment was performed again and the speed of rotation changed very early, according to a criterion described later (Figure 5). The difference between the two experiments (short and long steps, see Table 3) indicate that the loss of accuracy is negligible compared the reduction in duration. For example on the first step, the optimized approach gives an equilibrium saturation with a relative error of 4% (0.3 % of the whole saturation range) when duration cut is around 75%. From our experience, shortening the rotation steps affects neither the determination of T_2 nor the final saturation of the next step.

Table 3: numerical values corresponding to Figure 5. S_{fin} is the saturation measured at the end of the step. The errors induced when reducing the duration of the steps is negligible.

		Classical	Fast	Error
	Sfin	0.0808	0.073	4%
STEP 1	Seq		0.084	
	Duration	40 H	10 H	Gain: 75 %
	Sfin	0.1136	0.109	2%
STEP 2	Seq		0.116	
	Duration	20 H	15 H	Gain: 25%

Speed change criterion

The experiment described above indicates that the rotation step can be shortened considerably. The question is to determine the optimum measurement time. We propose a criterion based on the fluctuations of the extrapolated equilibrium saturation, replacing the (classical) criterion based on the fluctuations of the measured saturation.



Figure 6 : estimation of the true equilibrium saturation as a function of the measurement time in the case of the 2^{nd} drainage (data from Figure 3)

To illustrate such a criterion, we estimated the equilibrium saturation as a function of measurement time (Figure 6 and Figure 7) for the data plotted in Figure 2 and Figure 3. It is seen that the extrapolated saturation is fairly stable after a measurement time of about 6 hours for most of the steps. If one chooses a saturation accuracy target of about 1%, the speed of rotation can be changed when the fluctuations in the extrapolated saturation are smaller than 1% during 4 hours. Note that the extrapolation is only slightly dependent on the initial guess in the optimization routine. Consistent results are obtained when setting the initial equilibrium saturation to the actual saturation (default values for T_1 , T_2 and w are respectively 1 hr, 10 hr and 0.5 respectively).



Figure 7 : estimation of the true equilibrium saturation as a function of the measurement time in the case of the forced imbibition (data from Figure 2)

If applied to the experimental data from Figure 2 and Figure 3, the duration of the experiment could have been shortened by a factor of two. But the ability to provide good extrapolation is strongly dependent on the quality of the measurement system. For example, the data of step 6 in the imbibition experiment (Figure 2) were perturbed for an

unknown reason. As a consequence, the extrapolation is very uncertain at short time (Figure 7, curve 6) and the standard duration of 16 hr is necessary. When the measurement system is running at optimum conditions, the duration can shortened down to 6 hours.

Extrapolation using manual reading data

For non automated centrifuges (i.e. production is measured manually using a stroboscope), step duration cannot be shorten but accuracy can be increased by extrapolating equilibrium saturation. As shown in Figure 8, production curves measured on two different samples can be fitted satisfactorily, despite the loss of accuracy. However, care must be taken not to over weight data at short times and this occurs naturally because more data points are taken when variation are large. The last points are of primary importance because they impact directly on the curve behavior near equilibrium. Hence, the objective function in the optimization routine was build with weight coefficients, which depend on local data density.



Figure 8: Modeling of manual reading data improves the accuracy of equilibrium saturation. Sample 1: K=648 mD, Sample 2: K=80 mD.

Discussion

The choice of a speed change criterion must agree with the accuracy target of the user. This target also depends on the final use of the capillary pressure : wettability estimation, flooding interpretation, etc. The accuracy of the average saturation estimation directly impacts the calculation of capillary pressure but the lack of equilibrium is difficult to detect. Indeed, when calculating the capillary pressure curve from equilibrium average saturation, the standard procedure requires to back calculate the average saturation curve from the estimated capillary pressure curve and compare it to the experimental data. However, this procedure will only reveal strong data set inconsistency.

Another advantage of extrapolating the transient saturation curve is to obtain the T_1 and T_2 values. As described in the first section, these characteristic times have a physical significance. In the example of Figure 2 and Figure 3, it is striking to see that T_2 values are very similar from one step to another, as well as from drainage to imbibition cycle. This observation strongly suggests that T_2 may be a characteristic of the sample, related to the

relative permeability curves. We suspect that T_1 is related to the capillary pressure curve and therefore to the speed sequence.

CONCLUSION

A method is presented to extrapolate transient saturation curves measured during a multi-speed centrifuge experiment to obtain an accurate estimation of equilibrium saturation. The method uses a bi-exponential function comprising 4 parameters to fit the experimental data in order to obtain the saturation at infinite time.

The bi-exponential model is validated on data acquired during various cycles (airwater 1^{st} drainage, oil-water forced imbibition and 2^{nd} drainage) on various samples (sandstone, carbonate of intermediate wettability). Without extrapolation, the typical duration of a rotational step is 16 hr for a 100 mD sample and yields a total duration of a centrifuge cycle of about 5 to 7 days. Due to various influences (relative permeability and wettability, fluid system used), this duration can vary strongly. When applying the proposed extrapolation method during the experiment, it is possible to decrease the duration by at least a factor of two, depending on the experimental conditions and the accuracy of the measurement technique.

Further work will focus on the establishment of precise guidelines to set the duration of the rotational steps as a function of core length, absolute and relative permeability, measurement noise, etc.

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