Magnetic Resonance Imaging 56 (2019) 131–137

Contents lists available at ScienceDirect

Magnetic Resonance Imaging

journal homepage: www.elsevier.com/locate/mri

Original contribution

Fast and robust quantification of liquid inside thin fibrous porous materials with single-sided NMR

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ARTICLE INFO

Keywords:
Thin porous media
Single-sided NMR
Liquid quantification

ABSTRACT

Single-sided NMR with the NMR-MOUSE is employed for the characterization of fluids in fibrous and open foam materials. One of the key aspects of this study is the quantification of the fluid amount. To this end critical information was provided by a relaxation study. Using 2 mM/L of a Gd^{3+} relaxation agent the repetition time could be shortened to 250 ms, improving the correlation coefficient between liquid amount and signal amplitude from $R^2 = 0.893$ to $R^2 = 0.982$. To assess reproducibility and instrument precision, calibration experiments were repeated several times and their variation investigated. The results showed that the device is highly precise and robust with a standard deviation for liquid quantification of less than 1%.

1. Introduction

A major application area of NMR is the study of porous materials which started in the early days of NMR when Torrey investigated the complex diffusion dynamics of fluids inside porous media [1]. From the early 1970s on NMR relaxation properties have been used to characterize the permeability of porous rock [2], and the basic physics of surface relaxation and its relationship to the pore-surface area have been studied intensively [3]. Spin relaxation is still being used to probe the surface-to-volume ratio (SVR) [4]. Since then, NMR relaxation has been explored extensively in studying porous materials such as cement [5] and subsurface-rock formations [6]. However, fibrous substrates and in particular thin fibrous materials have received much less attention. A thin porous layer is defined by a thickness that is orders of magnitude lower than its lateral dimensions [24]. Such materials are employed in a wide range of products such as fuel-cells, paper, filters, fluid absorbents and barrier materials, textiles, diapers, and pads. The interaction of fluids with the fibers is of fundamental importance for the processing and function of these materials.

Magnetic resonance imaging (MRI) can monitor fluid distributions and flow through porous materials noninvasively. For example, MRI has been employed to determine the moisture content in textile and fibrous materials such as carpets [7], filtration materials [8,9] and paper [10,11]. However, quantitative studies and the generation of calibration curves with MRI methods can be challenging. Ideally, one would like to have MRI experiments for this purpose that produce images of the spin density only without other contrast parameters such as the relaxation times T_1 and T_2 and diffusion so that the signal intensity is proportional to the liquid amount. When using standard MRI sequences such as the spin-echo and the gradient-echo sequences, the T_1 weight can be eliminated from the image by setting the repetition delay sufficiently long. Signal attenuation from T_2 relaxation and diffusion can be minimized by choosing imaging sequences with short deadtime, for example, single point imaging (SPI) techniques [12], UTE [20,21], ZTE [22], and SWIFT [23]. However, the low sensitivity of MRI techniques, challenges the quantification of low liquid amounts in thin porous media.

Alternatively, mobile single-sided NMR devices can be used for determining liquid distributions inside porous materials, such as soil, concrete, building materials and food [13–15]. The Profile NMR-MOUSE is an example of such a sensor that can provide depth profiles with spatial resolution better than 10 μm due to a strong gradient perpendicular to the sensor surface and very low lateral-field variation at a fixed measurement distance from the sensor [16]. In this work, this type of sensor serves to measure relaxation signals to quantify liquid amounts. Other than imaging, most relaxation measurements are without spatial resolution, but such a single-sided probe can provide information on the liquid content of a sample as a function of the depth.

One of the key aspects of the study is the quantitative measurement of fluid amount in different thin, porous media by NMR relaxation with a single-sided NMR sensor. The measurements provided the critical information needed to quantify the liquid amount. To assess the

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https://doi.org/10.1016/j.mri.2018.09.022
Received 30 July 2018; Received in revised form 20 September 2018; Accepted 20 September 2018
0730-725X/

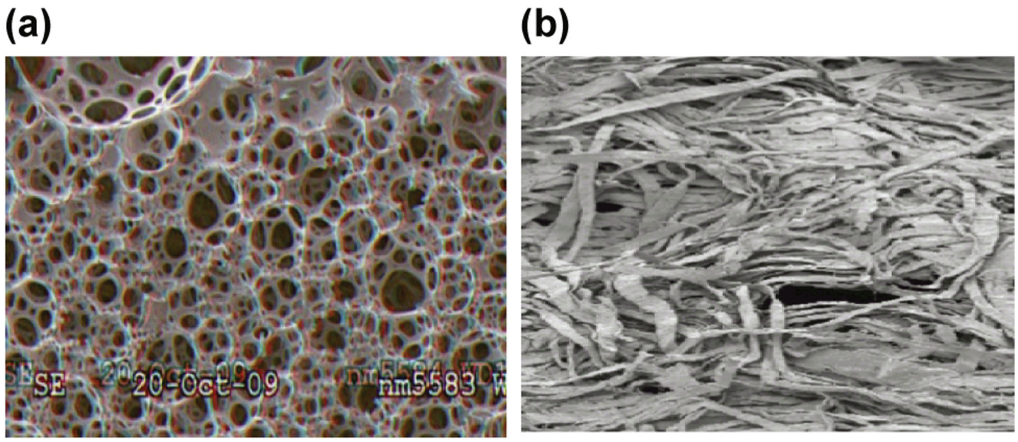

Fig. 1. (a) Microscopic structure of open foam material. (b) Microscopic structure of filter paper.

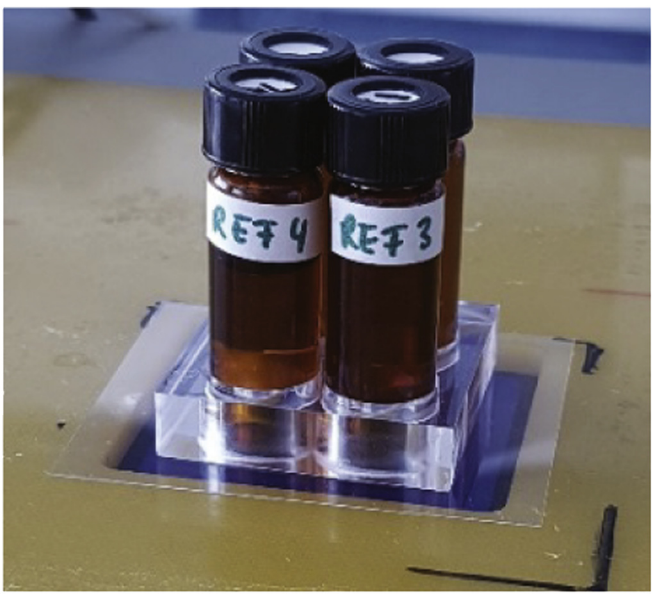

Fig. 2. Placement of the tubes on the NMR sensor for the Calibration tests.

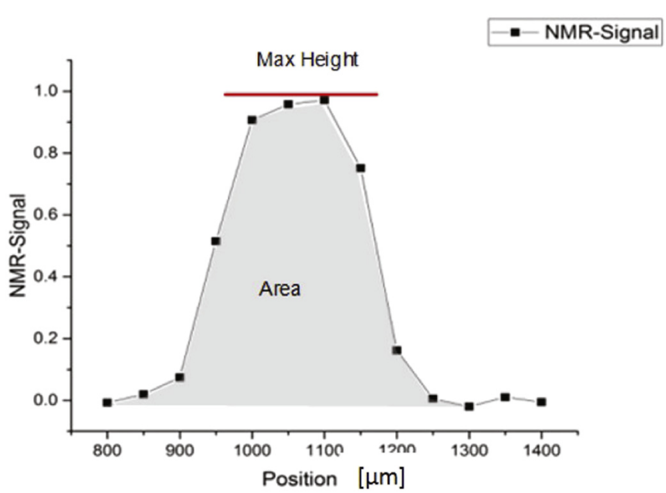

Fig. 3. Profile of a filter paper soaked with 40 µL 0.9% saline solution. To quantify the liquid load, the area under the curve and maximum height of the curve were investigated.

robustness of the quantification, the precision of measurement was determined by executing calibration experiments several times and determining their variation.

2. Experimental procedure

2.1. Instrument

The device that is used for the measurements was a PM-25 NMR-MOUSE from Magritek (Aachen, Germany). It has a depth range varying from 5 to 25 mm, sufficiently large to cover the thickness of different porous materials. The sensitive slice above the surface of the sensor is $4 \times 4\,cm^2$, and 1D profiles can be acquired across different depth ranges with the help of a high-precision lift. This device generates a static gradient of 8 T/m in the direction perpendicular to the sensor surface, and the protons in the sensitive slice are in resonance at a frequency of 13.5 MHz.

2.2. Relaxation measurements

Two different pulse sequences were used, a CPMG sequence for T_2 relaxation measurements (echo time 150 µs, number of echoes 4096, repetition time 6000 ms, number of scans 8), and a saturation-recovery sequence to measure T_1 relaxation (echo time 63.5 µs, number of echoes 256, repetition time 100 ms, number of scans 8, recovery time 9000 ms).

2.3. Liquid quantification

The proportionality of the NMR signal amplitude to the liquid amount was investigated with and without a relaxation agent. For the experiments without a relaxation agent, 200 µm thick WHATMAN filter paper was cut into $4 \times 4\,cm^2$ samples and placed on a glass slide on top of the NMR sensor. Different amounts 0.9% saline solution (40 µL, 80 µL and 160 µL) were dripped onto the filter paper from a pipette. The filter paper was covered by another glass slide to avoid evaporation during the measurements. A NMR profile with a 50 µm spatial resolution was measured through the filter paper sample using the CPMG sequence and the high-precision lift.

Whenever the fluid inside a porous material is not able to move freely, its relaxation is accelerated by contact with the confining structures [17]. In order to investigate this in more detail, a series of experiments was performed to determine the relaxation inside porous materials. T_1 and T_2 were measured for different saturation levels. Due to the fact that the proportion of bound and free fluid inside a porous structure is most important for our analysis, two materials differing widely with respect to their pore structure were investigated. First filter

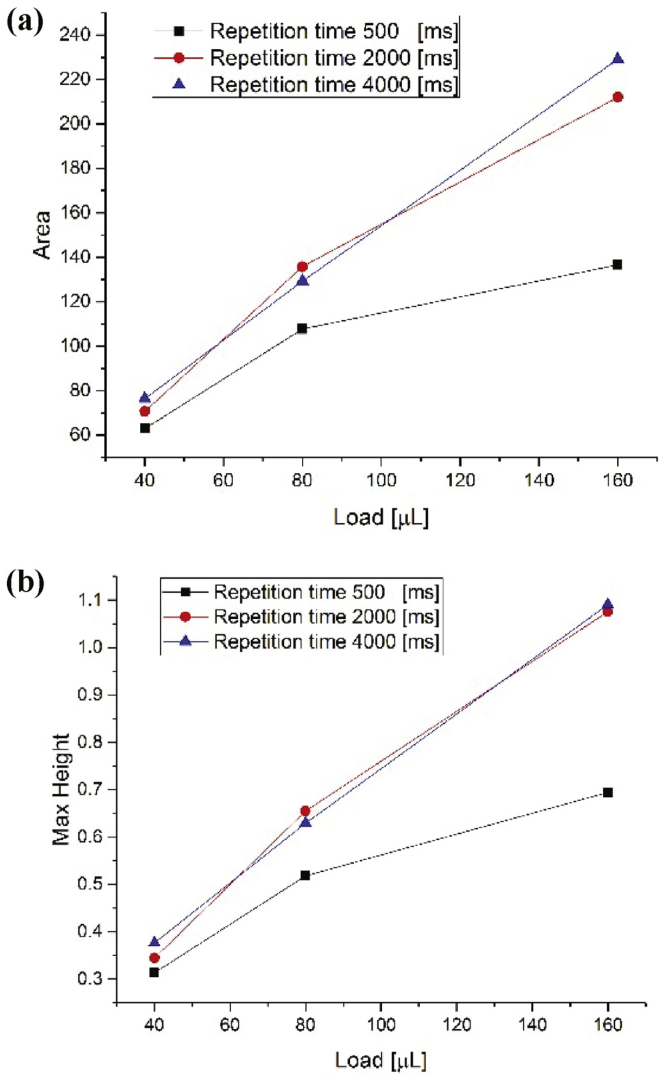

Fig. 4. Quantification of the 0.9% saline water load for different repetition times. (a) Area under the curve. (b) Maximum height for the profile of filter paper.

Table 1
Correlation coefficients for area and maximum height versus liquid load for different repetition times and correspondingly different acquisition times.

Repetition time [ms]	R^2 Area-Load	R^2 Max-Load	Measurement time for 1 mm at 50 µm resolution [min]
500	0,8083	0.8933	3
2000	0.9786	0.9946	11
4000	0.9995	0.9989	22

paper (FP) was used, which binds a considerable amount of fluid. Second, an open foam material was used, which contains mostly free fluid. The pores in the foam material are largely open and the material is hydrophobic so that the fluid inside the pores can be considered to be free fluid (Fig. 1a). The structure of filter paper consists of densely packed fibers, which absorb the fluid in the gaps and into the fibers. This fluid inside is expected to be partly bound (Fig. 1b).

Following the experiments without relaxation agent, experiments with relaxation agent were performed to study in how far the faster liquid relaxation affects the proportionality between signal and fluid amount. For these experiments 2 mM/L of diethylenetriamine pentaacetic acid gandolinium III dihydrogen salt hydrate (381667 Sigma-Aldrich) was added to decrease T_1 relaxation and thereby reduce the required acquisition time.

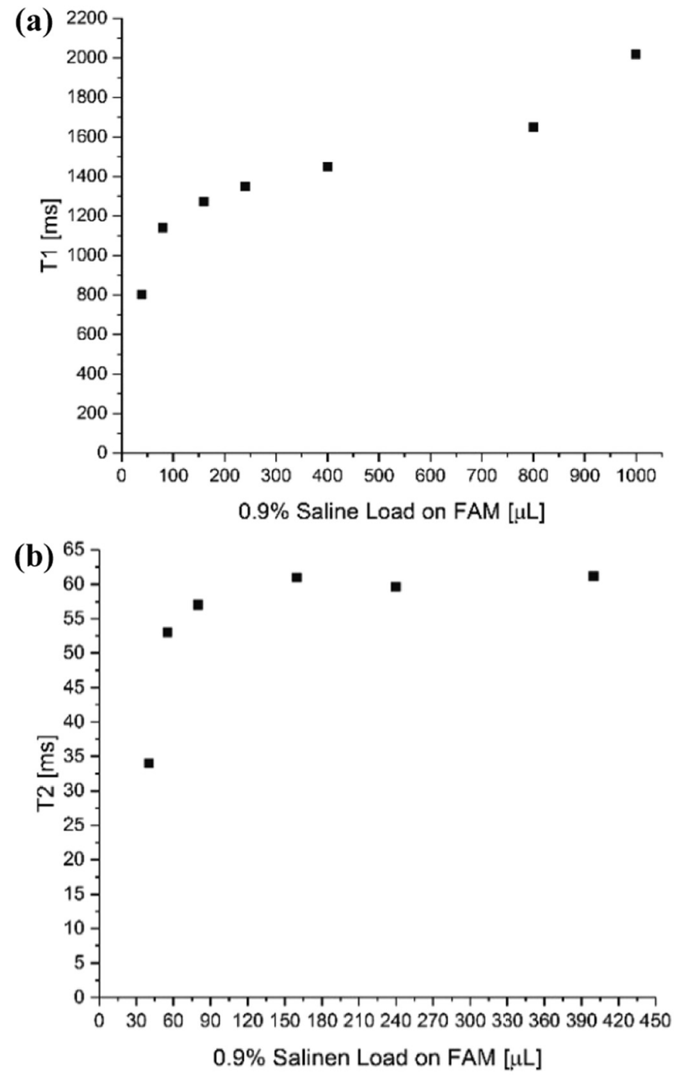

Fig. 5. Relaxation measurements for open foam material with different saturations of 0.9% saline solution. (a) T_1 measured with a saturation-recovery sequence. (b) T_2 measured with a CPMG sequence.

2.4. Calibration and precision of dynamic measurements

In the dynamic measurements the amounts of liquid passing through a fixed sensitive volume were measured with high temporal resolution (250 ms). Such measurements provide valuable information to understand the material and product behavior in the first few seconds after liquid application.

To calibrate the signal amplitude with the fluid amount in dynamic measurements four identical 12.3 mm diameter glass tubes were filled with 0.9% saline solution and placed vertically on top of the sensor (Fig. 2). The thickness of the sensitive volume was fixed to 400 µm, and the sensitive area of the PM-25 device is 4 × 4 cm^2. With these parameters, the liquid volume inside the sensitive volume was estimated from the known volume of each tube (48 µL).

In order to test the ability of the PM-25 sensor to monitor the dynamic behavior inside porous materials and products, the following experimental procedure was executed: First, all calibration tubes were placed into the sensitive volume. Every 30 s one of the tubes was removed while the NMR signal amplitude was measured continuously. This resulted in a step function that can be employed to calibrate the

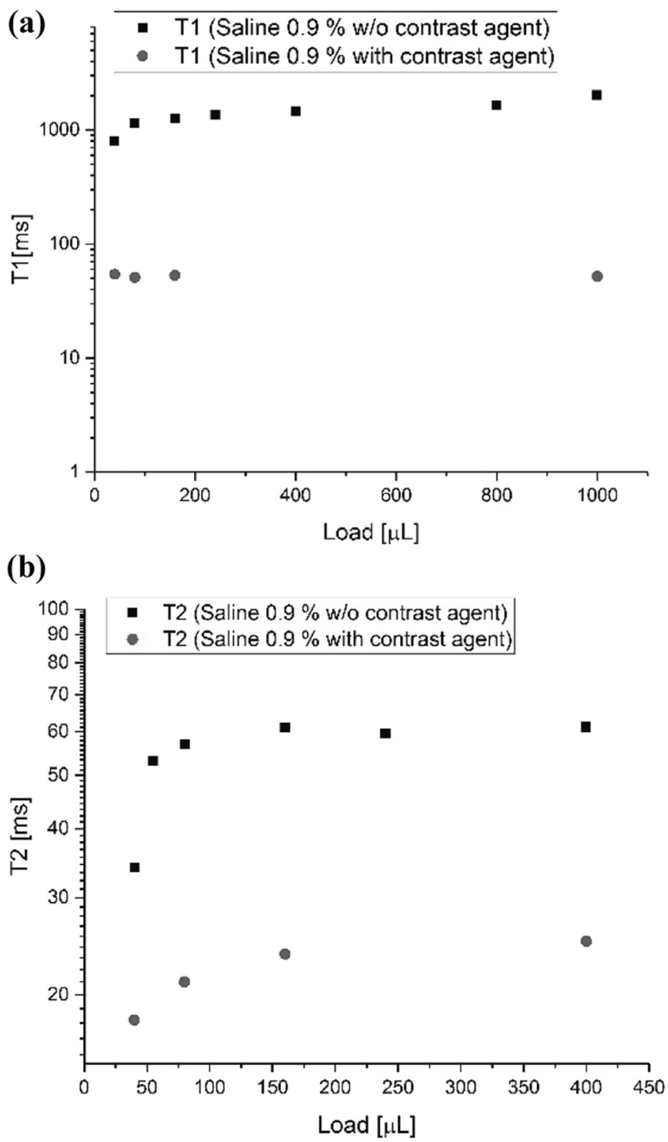

Fig. 6. Relaxation times for filter paper with different saturation levels of 0.9% saline solution. (a) T_1 relaxation. (b) T_2 relaxation.

liquid volume with the signal amplitude. The procedure was repeated five times with the same tubes to investigate the instrument precision and reproducibility.

2.5. Calibration and precision of profiling

Similar to the calibration procedure for dynamic measurements, four identical 1.23 cm diameter glass tubes filled with 0.9% saline solution served to calibrate the signal amplitude obtained from the sums of the first 8 echoes versus the liquid volume. In profiling measurements, the sensitive volume is moving through the sample with the help of a high-precision lift. A total range of 2000 μm along the tube's height and inside the tubes was profiled to evaluate the stability of the signal with a spatial resolution of 50 μm. The same experiment was performed with 4, 3, 2 and 1 tubes. Each experiment was repeated five times to investigate instrument precision.

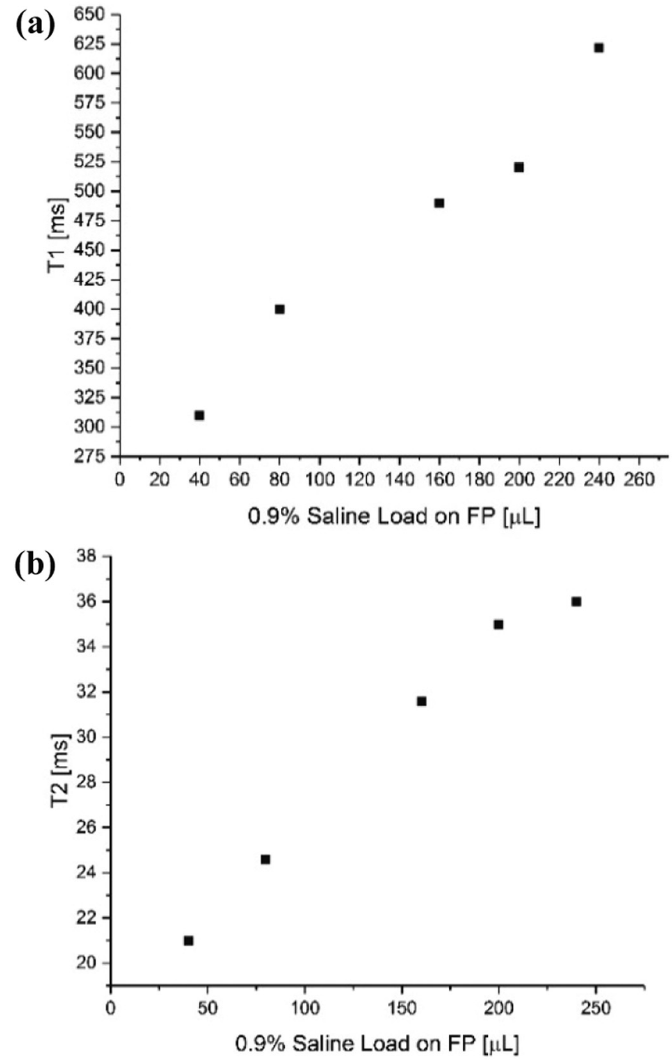

Fig. 7. Relaxation measurements for open foam material as a function of 0.9% saline solution load with and without relaxation agent (Gd^{3+} complex). The relaxation axes are logarithmic. (a) T_1. (b) T_2.

Table 2
Comparison of T_1 and T_2 relaxation times, correlation coefficients, and total experiment times for 0.9% saline solution with and without relaxation agent.

	T_1 Free fluid [ms]	T_2 Free fluid [ms]	R^2 area-load (TR:500 [ms])	R^2 max-load (TR:500 [ms])	Experiment time [min]
0.9% saline w/o relaxation agent	2160	50	0,8083	0.8933	3 (with 16 scans)
0.9% saline with relaxation agent	50	32	0.9761	0.9824	1.5 (with 8 scans)

3. Results and discussions

3.1. Relaxation and proportionality study

There are two possible options to quantify the liquid amount inside porous materials using the NMR-MOUSE (Fig. 3). First, one can use the area below the profile curve as the quantity to correlate with the liquid amount. Second, one can use the peak of the curve. Therefore, both values were determined for NMR profile measurements with three

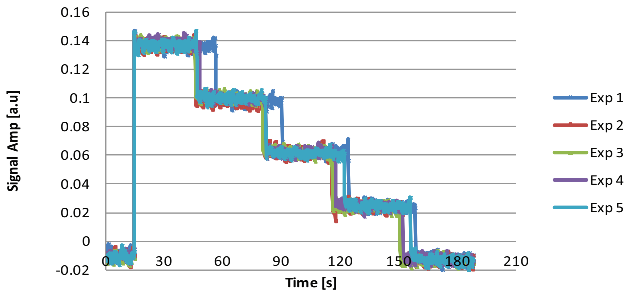

Fig. 8. Five repetitions of experiments for calibration with four identical glass tube containing 0.9% saline solution and 2 mM Gd^{3+} relaxation agent.

different amounts of added water and three different repetition times, 500, 2000 and 4000 ms (Fig. 4).

Fig. 4 shows that both the area and the maximum height strongly correlate with the amount of water in the sample. The correlation coefficients R^2 are listed in Table 1. Use of the area is physically justified and the correlation coefficient is slightly better than for the height. Therefore, the area below the curve was used for liquid quantification. The total acquisition time for each repetition time and for a 1 mm thick sample was calculated as

$$t = \frac{d}{Re} \times NS \times Rt \tag{1}$$

where t is the total acquisition time, d is the thickness to be scanned (1 mm here), Re is the spatial resolution used for profiling, NS is the number of scans per slice of profiling, and Rt is the repetition time for the CPMG sequence. Table 1 shows that the linearity as expressed by the correlation coefficient improves with increasing repetition time between the pulses. This is expected because an increasing repetition time allows for a more complete build-up of the thermodynamic equilibrium magnetization. For sufficiently long repetition times, the signal amplitude is no longer T_1-weighted. Additionally, in all these experiments the signal amplitude was estimated from the sum of the first eight echoes, which is less T_2 weighted and essentially depends only depends on the proton density.

The T_1 and T_2 relaxation times measured for the free 0.9% saline solution are 2166 ms and 62 ms, respectively. Fig. 5 shows the measured T_1 and T_2 relaxation times for open foam material in the case of 0.9% saline solution as fluid. The value for T_1 increases with increasing saturation. For the highest load, it reached 2100 ms, which is similar to the value of free saline water (Fig. 5a). When the open foam is fully saturated, the protons relax in the same way as in the free fluid. This suggests that most liquid inside foam is free and not bound. These results are fully consistent with the expectations about the open foam material.

In order to avoid T_2-weighting of the signal amplitude, T_2 should be high enough so that the signal does not decay during the first eight echoes. The results of the T_2 measurements (Fig. 5b) demonstrate that T_2 is high enough for adequate quantification, even at low saturation (34 ms). The echo time used in the CPMG sequence was 0.15 ms, and thus the time required for acquisition of four echoes was 0.6 ms. This is far less than T_2.

Furthermore, the T_2 values reach their maximum value at low

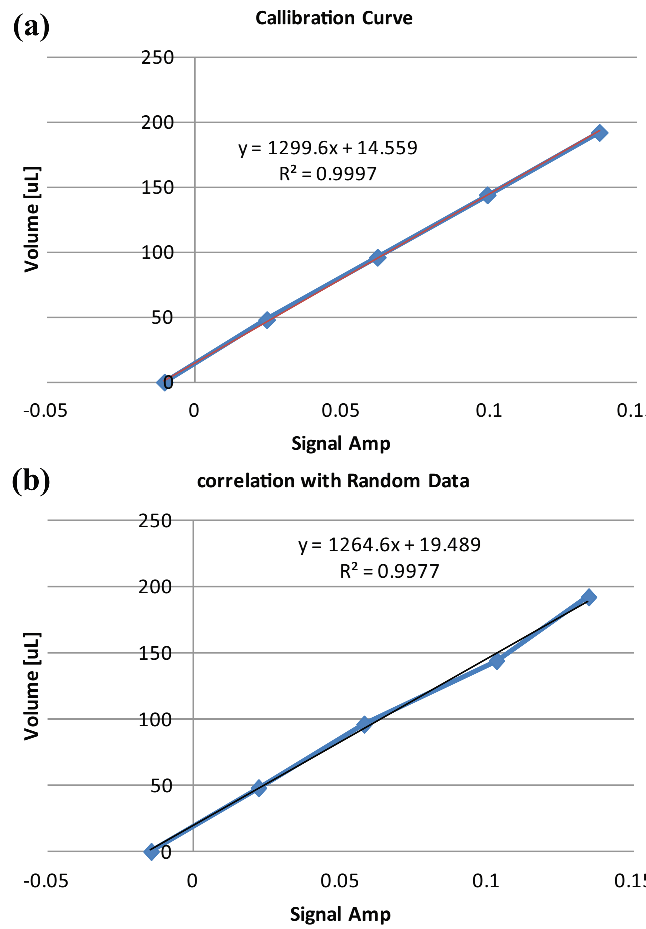

Fig. 9. (a) Calibration curve for dynamic measurement, 0.9% saline solution containing 2 mM Gd^{3+} with averaged data. (b) Calibration with a random set of data from all experiments to study the effect of averaging on linearity.

amount of saturation and do not change significantly until the foam is fully saturated. The load-dependent trend for the relaxation times confirms that the foam contains largely free liquid even at lower loads.

The same measurements were executed with filter paper. The trend of T_1 (Fig. 6a) indicates an increase in relaxation time with increasing saturation. For fully saturated filter paper with (240 μL saline solution), T_1 = 622 ms. This relaxation time is short compared to that of the free fluid and saturated foam material, indicating a high amount of bound liquid inside the porous structure of the filter paper. Fig. 6b shows the T_2 relaxation times for different saline-solution loads on filter paper. T_2 was found to be 20 ms at low saturation and 36 ms at full saturation.

Fig. 7 shows the T_1 and T_2 relaxation times with and without the presence of relaxation agent for a foam sample at different liquid loads. When comparing the T_1 relaxation times for liquid with and without relaxation agent, it is observed that the relaxation agent strongly reduces T_1 to values as low as 50 ms independent of the saturation level of the filter paper. This observation suggest that the repetition time and therefore the total acquisition time can significantly be reduced by

Table 3
Correlation and instrument precision for dynamic measurements tested with 0.9% saline solution containing 2 mM Gd^{3+} relaxation agent.

	Volume [μL]	Max value	Min value	Average signal	Variance	Standard deviation [%]
4 tubes	192	0.1382	0.13634	0.1372	6.97 E-07	0.0835
3 tubes	144	0.1008	0.0974	0.099	2.09 E-06	0.1446
2 tubes	96	0.0626	0.0613	0.0621	2.14 E-07	0.0462
1 tubes	48	0.02481	0.0242	0.0246	5.50 E-08	0.0234
Base	0	−0.0083	−0.0113	−0.0100	1.36 E-06	0.1168

Table 4
Results of the correlation and instrument-precision study for profiling measurements with 0.9% saline solution containing 2 mM Gd^{3+} relaxation agent.

	Volume [μL]	Max signal	Min signal	Average signal	Variance	Standard deviation %	Max area	Min area	Average area
4 tubes	960	0.2698	0.2656	0.2680	2.52E-06	0.1589	539.52	531.21	536.06
3 tubes	720	0.2027	0.1991	0.2005	2.40E-06	0.1550	405.49	398.27	401.08
2 tubes	480	0.1340	0.1314	0.1330	1.26E-06	0.1126	267.92	262.87	266.00
1 tube	240	0.0672	0.0638	0.0655	1.6 E-06	0.1281	134.49	127.57	130.93

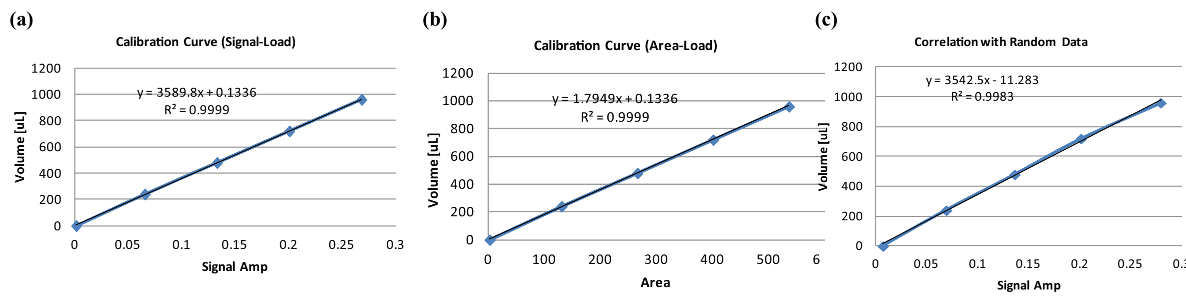

Fig. 10. Calibration curve for profiling measurement using (a) signal amplitude, (b) the area below the profiling curve, and (c) a random selection of data from all experiments to study the effect of averaging on linearity.

relaxation agents to $5T_1 = 250$ ms for the dynamic measurements. However, in profiling measurements we have used repetition a time of 500 ms, which is about $10T_1$ so that all magnetization will build up in the time between consecutive scans.

To check the proportionality of the NMR response with the liquid load amount, profiles of three open foam samples with three different loads of 0.9% saline solution and the Gd^{3+} complex were measured with a CPMG sequence. Again, the area and the maximum height of each profile curve were used to correlate with the liquid load amount. The resulting correlation coefficients are listed in Table 2 together with the correlation coefficients obtained with the use of the relaxation agent.

According to Table 2, T_1 relaxation is reduced to 50 ms by the relaxation agent. Thus, high linearity of the calibration curve can be assured even when at a repetition time as short as 500 ms. The correlation coefficients for area and maximum height against the load are 0.9761 and 0.9824, respectively. Due to the high sensitivity that could be reached with the relaxation agent, only 8 scans were acquired. Therefore, the total experiment time was reduced to 1.5 min for scanning a depth range of 1 mm with a spatial resolution of 50 μm. However, it worth mentioning that the correlation coefficient is high also with longer repetition times (see Fig. 4). Longer repletion times could be used when it is not possible to employ a relaxation agent for experimental reasons.

3.2. Calibration and precision study

Fig. 8 shows the results obtained for five experiments executed with the same sample tubes containing 0.9% saline solution and 2 mM/L Gd^{3+} relaxation agent. The step sizes for the signal amplitude correlate to the volume of 48 μL per tube inside the sensitive slice.

The signal amplitudes within each 30 s time step were averaged for each experiment, and the associated standard deviations and variances were determined (Table 3). The average values derived from these five experiments were fitted to the liquid amount in a calibration curve (Fig. 9a). The five-point calibration curve fits a line with a correlation coefficient of $R^2 = 0.9997$. Furthermore, standard deviation is smaller than 1% in all the experiments, indicating indicated that the instrument precision is very high.

In order to assure that averaging of data in each experiment does not affect the variation of the test response, the quality of the calibration is also evaluated using randomly chosen data, i.e. one signal amplitude for 1, 2, 3 and 4 tubes selected randomly from the repeat experiments (Fig. 9b). The linear fit and the resulting correlation coefficient are almost the same. This suggests that averaging of the signal amplitude in 30 s windows and in each experiment for different volumes does not have a meaningful effect on the calibration curve and function.

The calibration procedure for the profiling measurements was performed with the same glass tubes and the same liquids that were used for the calibration of the dynamic measurements. The average of the signal amplitude in each profile has been taken for calibration of the relationship between signal intensity and liquid volume. In addition, the corresponding standard deviations and variances have been calculated (Table 4). The calibration curve (Fig. 10a) has a high correlation coefficient of $R^2 = 0.9999$ with the experimental data.

In addition to signal amplitude, the area beneath the profiling curve was taken for calibration of the liquid volume (Fig. 10b). In order to make sure that averaging of data in each experiment for signal does not affect the variation of the test response, the calibration was also tested with randomly chosen signal amplitudes for 1, 2, 3 and 4 tubes (Fig. 10c). Results show that, the linear fit and the resulting correlation coefficient are almost the same. This suggests that averaging of the signal amplitude does not have not significantly improved the precision of the calibration.

4. Conclusion

In this paper, a single-sided NMR sensor, the NMR-MOUSE, was employed to quantify the liquid amount inside porous materials. In order to understand the effects of free and bond fluids in the material on the relaxation times, relaxation was measured on filter paper where most of the fluid is bound and on an open foam material where most is free due to its hydrophobic nature and large pores. In addition, a relaxation agent with minimum effect on liquid and material properties was introduced to further decrease the repetition time and thereby the total experiment time. The use of 2 mM/L of Gd^{3+} complex and a repetition time of 500 ms resulted in an improvement of the correlation coefficient between liquid amount and signal amplitude from $R^2 = 0.893$ to $R^2 = 0.982$.

A series of experiments was performed to investigate the instrument precision and to correlate the signal amplitude to the actual liquid amount in both profiling and dynamic measurements. Results showed that the device is highly precise with a standard deviation less than 1%

for both types of measurements.

The single-sided low-field NMR sensor has already been recognized as a powerful tool to investigate the interaction of fluid and thin fibrous porous materials and to validate numerical studies [18,19]. Therefore, establishing a robust and fast quantitative measurement of liquid distributions will further provide valuable insights into fluid transport in porous media.

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