

Porous media characterization by PFG and IMFG NMR

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Abstract

Fully and partially filled with tridecane quartz sand was studied by different NMR techniques. The set of NMR experiments was carried out to obtain information about porous media geometry and fluid localization in it in case of partially filled porous space. The study was done using three NMR approaches: pulse field gradient NMR (PFG NMR), DDif experiment and tau-scanning experiment. The possibility to use all three approaches to study porous media properties even at the high resonance frequency is shown together with complementarity of the given by them information. Thus, first two approaches give information about porous sizes and geometry, at the same time tau-scanning experiment allows us to obtain information about distribution of internal magnetic field gradients in the porous space and draw conclusions about fluid localization in it.

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1. Introduction

Nuclear magnetic resonance (NMR) is one of the powerful techniques to study porous media properties [1–13]. Classical NMR techniques of porous media morphology and geometry study are based, mostly, on the use of external magnetic field gradients [7–13]. In this case the features of the translational mobility of the fluid molecules contained in the porous space give information about porous media characteristics.

Today a new methodical approach is being included in the NMR study of porous systems [13–15]. This approach uses internal magnetic field gradients (IMFGs) that appear in the porous media near the porous media–diffusant molecules interface due to magnetic susceptibility difference between them. From the classical NMR (diffusometry and relaxometry) point of view, appearance of additional unaccounted magnetic field gradients leads to the wrong interpretation of obtained experimental data and demands developing of special pulse sequences to minimize IMFG

contribution. On the other hand, internal fields and their gradient distribution are determined by the porous space morphology and, thus, could be used to obtain information about porous media itself. Development of the new NMR approaches based on the IMFG has obvious perspectives.

By now one experimental NMR technique has been developed by Song [15–17] that uses internal fields. The author calls the technique “diffusion decay in the internal field” (DDif). This technique is based on obtaining the information about some time-dependent function of IMFG averaging due to self-diffusion of fluid molecule confined to the porous media. The problem of internal field in porous media is discussed in a number of theoretical works also [18–20], but reasonable data are obtained only for the model systems (sphere pack, regular geometry porous space, etc.). In the present work, we continue our study of IMFG distribution in porous media by the technique we called tau-scanning experiment [4] that will be described under methods in detail.

The aim of the work is to carry out comparative data analysis of the results of three different approaches of porous media study by NMR, including 13-interval PFG pulse sequence together with techniques based on the IMFG.

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2. Materials and methods

Porous media structure and fluid localization in it were studied on a sample of quartz sand, with the particle size 90–100 μm , filled with tridecane. The study was done on fully and partially (20%) filled porous media.

Three NMR techniques were used: classical PFG NMR approach with 13-interval pulse sequence [10], DDif experiment [16] and tau-scanning experiment [4]. All studies were done on the NMR diffusometer with the proton frequency 300 MHz, maximum value of the external magnetic field gradient (in case it was applied) being 30 T/m.

Thirteen-interval pulse sequence is widely used for NMR porous media study due to its effect of partial IMFG compensation. The sequence is described in detail in [10] and is used in the present work to obtain information about porous media characteristics by the classical PFG NMR approach. The idea is to obtain diffusion decays in the wide range of diffusion times and to analyze $D_s(t_d)$ dependence of the average self-diffusion coefficient (SDC) on diffusion time. The features of $D_s(t_d)$ behavior provide information about porous sizes and porous space permeability [4,8].

DDif is a novel NMR technique of porous media characterization introduced by Song [16]. The idea is to measure the pore size length scale by the internal magnetic field in porous materials, since the magnetic field inhomogeneity reflects the underlying pore geometry [16]. The technique is developed to characterize nuclear spin magnetization decay due to diffusion in the internal field (DDif) and obtain pore length scale in terms of the diffusion behavior. Echo signal is measured by stimulated echo pulse sequence with diffusion time t_d as varying parameter ($t_d = \Delta - \frac{1}{3}\tau$, Fig. 1). The dependence $E(t_d)$ of the echo amplitude on the diffusion time is obtained in such an experiment. In order to calibrate the effect of the T_1 relaxation, the reference signal is obtained by a slightly modified sequence [16] and $E(t_d)$ is normalized on it. Such factorization result is some $A(t_d)$ dependence, which reflects “pure” diffusion decay in internal field and is characterized by plateau phase with signal intensity equal to zero, on the diffusion time when translational displacements of fluid molecules in porous media are comparable with pore size scale in the fully filled porous media [16]. Thus, DDif technique allows us to use IMFG to obtain information about porous sizes.

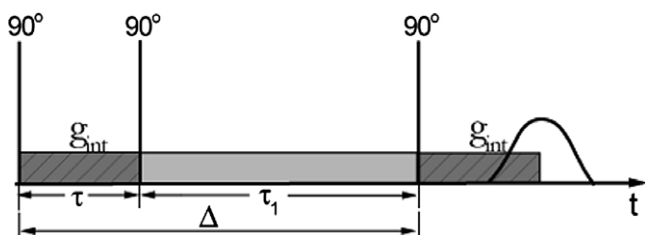


Fig. 1. Stimulated echo pulse sequence.

In the present work, the value of τ interval (Fig. 1) was set to $\tau = 2$ ms.

The third NMR technique was tau-scanning experiment proposed by our group [4]. The idea of the technique is to obtain information about IMFG distribution in porous media by carrying out an experiment that is in some sense opposite to the PFG NMR. In the classical diffusion experiment prescribed parameter is the amplitude of the applied external magnetic field gradient (g) and sought parameter is the value of self-diffusion coefficient that could be calculated from the experimental $A(g)$ dependence of echo amplitude on the applied magnetic field gradient. In the proposed tau-scanning experiment known parameter is self-diffusion coefficient of fluid molecules and sought one is the IMFG value. Tau-scanning experiment is also based on the stimulated echo pulse sequence (Fig. 1). Varying parameter is time interval τ between first and second, and third and echo, RF pulses, respectively. Experimental $A(\tau)$ dependence of stimulated echo amplitude on the τ value contains information about apparent IMFG distribution that diffusant molecules probe during given diffusion time in the studied porous media. The dependence could be written as:

$$A(\tau) \propto \int \Phi(g_{\text{int}}) \exp(-\gamma^2 \tau^2 g_{\text{int}} t_d D_s) dg_{\text{int}}, \quad (1)$$

where $\Phi(g_{\text{int}})$ is the apparent g_{int} (IMFG) distribution in the porous space that corresponds to the molecule displacements during given diffusion time t_d , where $t_d = \tau_1 + \frac{2}{3}\tau$, D_s is the average SDC of diffusant molecules in porous media.

Information about IMFG distribution could be derived from Eq. (1). In the rough approximation (as we have done in [4]) one can do it by replacing the integral with the sum of components. In this case IMFG distribution can be introduced as a histogram where the column height corresponds to the value of IMFG and the column width corresponds to the share of molecules probed this IMFG during given diffusion time [4]. The order of columns could be determined on the assumption that maximum internal gradient is localized near the porous wall. Thus, columns can be arranged in the order of IMFG value increasing from left to the right. It is reasonable to assume also that IMFG values decrease towards the pore center meaning that at the proposed histogram representation right part of histogram conventionally corresponds to the pore wall and left one to the pore center. As a label of x -axis for such histogram we chose “relative population” having in mind the above-given definition of column widths.

In the present work, Laplace inversion was used to find the solutions of Eq. (1) much as it is used to obtain SDC distributions from the diffusion experiments [21] for one-dimensional case. Data processing was done using the modification of the patented by Schlumberger R&D Laplace Inversion Procedure. All limits and technical questions are discussed widely in the papers of Hurlimann group [17,21–24]. We modified only the kernel in the scheme as consistent with the sequence and experimental

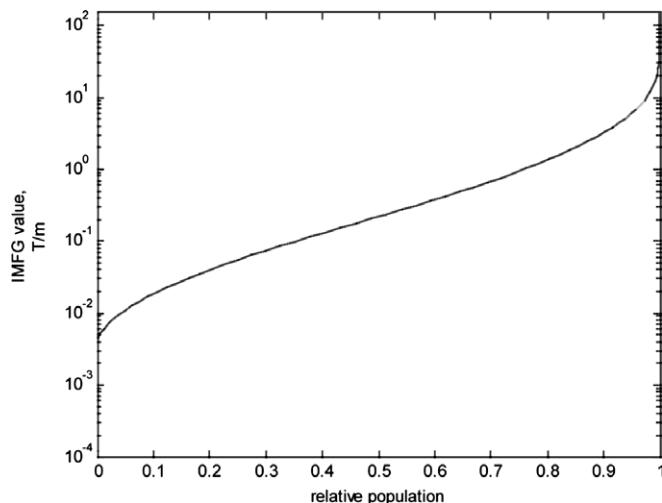


Fig. 2. IMFG distribution in the quartz sand fully filled with tridecane at the diffusion time equal to 38 ms.

condition we use. The regularization parameter α was equal to 10. An additional proof of the correct data treatment was the reasonableness of the inversion results in terms of their behavior with diffusion time (short and long diffusion time limits).

The obtained after inversion procedure data present $2 \times N$ matrix of the IMFG values and their populations (share of molecules probed certain IMFG value during the given diffusion time). Laplace inversion procedure seems to be a more correct way to analyze experimental data. It provides also required smoothness of the IMFG distributions in comparison with histogram and at the same time data can be represented in the same manner as histogram. In Fig. 2, IMFG distribution derived by Laplace inversion is shown for the fully filled quartz sand at the diffusion time equal to 38 ms.

Diffusion time in the tau-scanning experiment is the parameter that determines “spatial resolution” with which apparent IMFG values could be defined. The genuine distribution of IMFG in the porous media corresponds to the infinitely small diffusant molecule translational displacements (infinitely small diffusion time). Thus, carrying out tau-scanning experiment at the small diffusion times allows us to obtain information about IMFG distribution in porous space close to genuine one. At the same time, experiments at the different diffusion time values give information about the process of IMFG diffusion averaging.

3. Results and discussion

3.1. Fully filled with tridecane quartz sand

3.1.1. Classical PFG NMR

Thirteen-interval pulse sequence was used to obtain information about porous media structure by classical PFG NMR approach. $D_s(t_d)$ dependence of average SDC

on the diffusion time was analyzed in the range of diffusion times from 3 to 900 ms.

Talking about classical PFG NMR approach of porous media study, we should explain its features. In Fig. 3a, classical dependence $D_s(t_d)$ for the porous media characterized with single pore size is schematically shown. As one can see from the figure, three zones could be clearly distinguished: free diffusion zone $D_s = D_0 = \text{const.}$ in the area of short diffusion times, zone of the partially restricted diffusion $D_s(t_d)$ in the area of medium diffusion times and zone of averaged over the porous space diffusion $D_s = D_\infty = \text{const.}$ in the long diffusion time regime. Information about pore size could be obtained from the dependence using the approach offered in [8]. The idea is to calculate new dependence on the diffusion time of some effective SDC according to the following equation:

$$D_s^*(t_d) = \frac{D_0(D_s(t_d) - D_\infty)}{(D_0 - D_s(t_d))}, \quad (2)$$

where D_0 is the SDC of a bulk fluid, $D_s(t_d)$ is experimentally obtained dependence, and D_∞ is the SDC of fluid molecules in the long diffusion time regime. The main feature of the $D_s^*(t_d)$ dependence is its inverse proportionality to the diffusion time ($D_s^*(t_d) \propto t_d^{-1}$), that allows one to use Einstein equation to determine average restriction size as follows:

$$\xi = \sqrt{r^2} = \sqrt{6 \cdot t_d \cdot D_s^*(t_d)}. \quad (3)$$

In Fig. 3b, experimentally obtained $D_s(t_d)$ dependence is shown for the fully filled quartz sand. As one can see from the figure, the dependence is characterized by two (instead of one in classical case) regions with SDC dependence on the diffusion time separated by plateau area. Two regions with diffusion time dependence existence indicate two different restriction sizes presented in the studied porous space. To find corresponding restriction sizes we used the above-described (Eq. (2)) procedure twice by turns for both areas with $D_s(t_d)$ dependence. In the short diffusion time area $3 \text{ ms} > t_d > 40 \text{ ms}$ values $D_0 = 6.9 \times 10^{-10} \text{ m}^2/\text{s}$ and $D_\infty \equiv D' = 4.68 \times 10^{-10} \text{ m}^2/\text{s}$ were used for calculations. At the same time $D_0 = 6.9 \times 10^{-10} \text{ m}^2/\text{s}$ and $D_\infty = 4.0 \times 10^{-10} \text{ m}^2/\text{s}$ values were used for the long diffusion time regime. Restriction sizes were calculated using Eqs. (2) and (3) and were found to be equal to $\xi_1 = 5.6 \mu\text{m}$ for short diffusion time and $\xi_2 = 29 \mu\text{m}$ for long diffusion time regimes. Calculated numbers indicate that SDC dependence on the diffusion time in the area $t_d > 300 \text{ ms}$ corresponds to the restrictions constructed by the porous granules with the size 90–100 μm , at the same time $D_s(t_d)$ dependence on the short diffusion times is caused by microporosity.

Thus, classical PFG NMR approach allowed us to find out that studied porous media are characterized at least by bimodal distribution of porous sizes. Note that microporosity share is about 30 vol.% in this case (could be calculated from the D'/D_0 ratio). We assume that microporosity

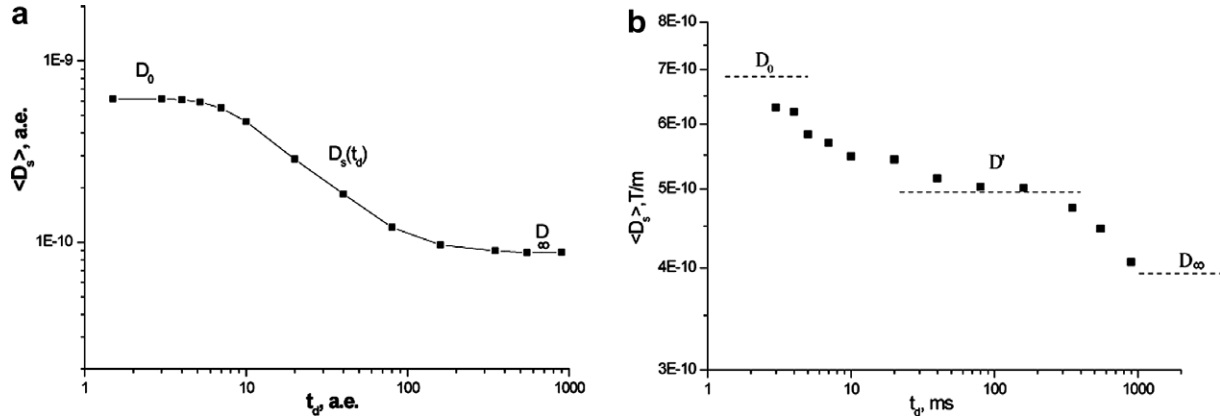


Fig. 3. $D_s(t_d)$ -dependences: (a) scheme of classical dependence for the single pore size, (b) experimental data for the 100% filled quartz sand.

is caused by the shape of porous media particles: microscopic study indicates that granules have a strongly asymmetric shape with large amount of chips on the surface.

3.1.2. DDif experiment

DDif experiment was used to obtain information about translational displacement of diffusant molecules required for the complete IMFG diffusion averaging. In Fig. 4, normalized dependence on diffusion time is shown for the fully filled quartz sand. As one can see from the figure, normalized dependence does reach plateau area $A(t_d) = 0$ at some characteristic time $t_d = t_d^0 = 430$ ms. As soon as average SDC $D_s(t_d^0)$ corresponding to that time is known, one can calculate translational displacement ξ^* required for the complete IMFG diffusion averaging. For the studied case it was found to be equal to $\xi^* = \sqrt{6 \cdot t_d^0 \cdot D_s(t_d^0)} = 35 \mu\text{m}$. Calculated value is in a good agreement with the macroporosity size obtained by PFG NMR. The result indicate that complete IMFG averaging in the fully filled porous space does happen on the distances about porous size.

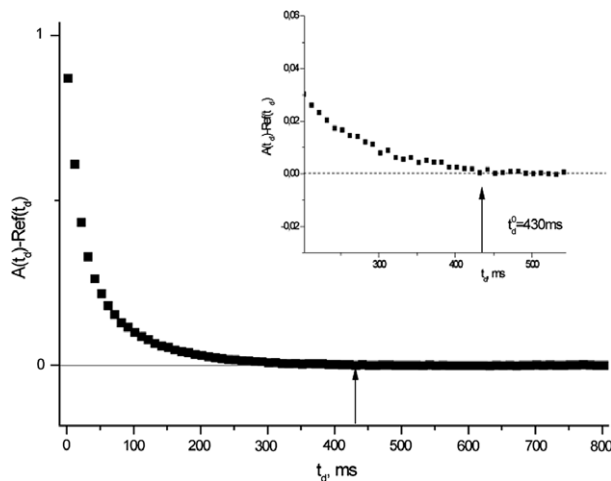


Fig. 4. $A(t_d) - \text{Ref}(t_d)$ for tridecane in fully saturated quartz sandstone.

3.1.3. Tau-scanning experiment

Tau-scanning experiment at different diffusion times was used to obtain information about IMFG distribution in porous space. Note, that obtained IMFG distributions are somewhat apparent once because they reflect information about IMFGs that are averaged over the translational displacements of diffusant molecules during certain diffusion time and genuine IMFG distribution in the porous media corresponds to the infinitely small diffusion time. Our hardware allows us to set minimum diffusion time equal to 5 ms that corresponds to the translational displacements about $4.5 \mu\text{m}$ for tridecane ($D_0 = 6.9 \times 10^{-10} \text{ m}^2/\text{s}$). In Fig. 5, IMFG distributions for fully filled quartz sand obtained by tau-scanning experiment are shown in the range of diffusion times from 5 to 320 ms. As one can see from the figure, distributions are different at different diffusion times and clearly demonstrate diffusion IMFG averaging. Detailed analysis of IMFG distribution features at different diffusion times was done.

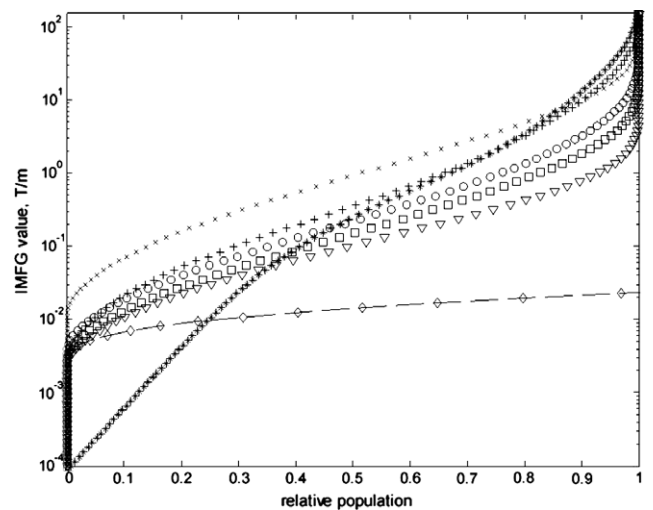


Fig. 5. IMFG distributions in fully filled quartz sand at the diffusion times 5 ms (stars), 10 ms (pluses), 20 ms (x-marks), 38 ms (circles), 80 ms (squares), 157 ms (triangles) and 320 ms (diamonds with dashed line).

At first, we should note that the largest IMFG value corresponds to the shortest diffusion time (about 200 T/m at 5 ms) and they decrease sufficiently with the diffusion time increasing. As one can see from Fig. 5, diffusion time increase from 5 to 157 ms leads to the decreasing of maximum IMFG value of about 1.5 orders of magnitude. Obtained IMFG distributions indicate also that distribution of internal fields is not parabolic (parabolic distribution of internal fields corresponds to the linear distribution of IMFG).

Second, IMFG distribution shape change with time shows possibility to distinguish two time stages of IMFG diffusion averaging process. First stage corresponds to the diffusion time from 5 to 20 ms: as one can see from Fig. 5, in this stage diffusion time increase leads to the maximum apparent IMFG values (right part of the IMFG distributions) that decrease together with the increase of apparent minimum IMFG values (left part of the IMFG distributions). Thus, the first stage of the diffusion IMFG averaging represents some local “blotting” of genuine picture of IMFG distribution.

Second stage of the diffusion IMFG averaging happens at the diffusion times longer than 20 ms up to complete averaging. As one can see from Fig. 5, in this stage both high and low apparent IMFGs become smaller with diffusion time increase and at the diffusion time 320 ms (diamonds with dashed line) they tend to have zero meaning. Thus, in the second stage macroscopic averaging happens all over the pore space and leads to the IMFG averaging to zero (complete averaging).

3.2. Partially (20%) filled with tridecane quartz sand

Partially filled porous space (20% filling with tridecane) was studied to investigate the possibility to determine fluid localization in the porous space by NMR.

3.2.1. Classical PFG NMR

In Fig. 6, $D_s(t_d)$ dependence for the partially filled quartz sand is shown. As one can see from the figure, it

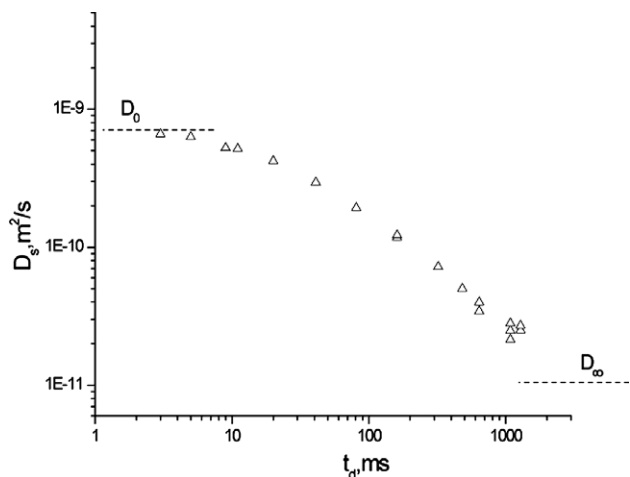


Fig. 6. $D_s(t_d)$ -dependence in partially (20%) filled sample.

is classical dependence for the system with one restriction size. Values $D_0 = 6.9 \times 10^{-10} \text{ m}^2/\text{s}$ and $D_\infty = 1 \times 10^{-11} \text{ m}^2/\text{s}$ were used to calculate restriction size using Eqs. (2) and (3). The size that was found to be equal to $\xi_r = \sqrt{r^2} = \sqrt{6 \cdot t_d \cdot D_s^*(t_d)} = 10 \text{ }\mu\text{m}$.

Note that D_∞/D_0 ratio characterizes permeability of the system in some sense. In our case this ratio is very low indicating small amount of contacts between areas with restricted diffusion.

Findings allow us to conclude that in the partially filled quartz sand fluid is localized in some areas with average size of about 10 μm characterized by restricted diffusion. It should be noted that found area size has the same order of magnitude with the microporosity ($\xi = 5.6 \text{ }\mu\text{m}$) which was found in a fully filled sample.

3.2.2. DDif experiment

DDif experiment was under our high interest to study features of diffusion averaging in the partially filled porous space. Note, that IMFG origin in the porous space is the magnetic susceptibility difference between porous material and fluid molecules, at the same time fluid molecules are markers themselves and in the fully filled porous space complete IMFG averaging is determined by pore size. What would be a characteristic distance of IMFG averaging in the partially filled porous space?

In Fig. 7, normalized on the T_1 relaxation contribution echo amplitude dependence on diffusion time in DDif experiment is shown for the partially filled quartz sand.

As one can see from the figure, $A(t_d)$ dependence does reach a plateau indicating that IMFG averaging happens in the partially filled porous space. In the studied case it was found that characteristic diffusion time of the process is equal to $t_d = t_d^0 = 310 \text{ ms}$. Note that this diffusion time is smaller by comparison with the corresponding one for the fully filled porous media. Calculated scale of complete IMFG averaging in case of partially filled sample was found to be $\xi^* = \sqrt{6 \cdot t_d^0 \cdot D_s(t_d^0)} = 12 \text{ }\mu\text{m}$ that is sufficiently smaller than the macropore size. The result indicates that

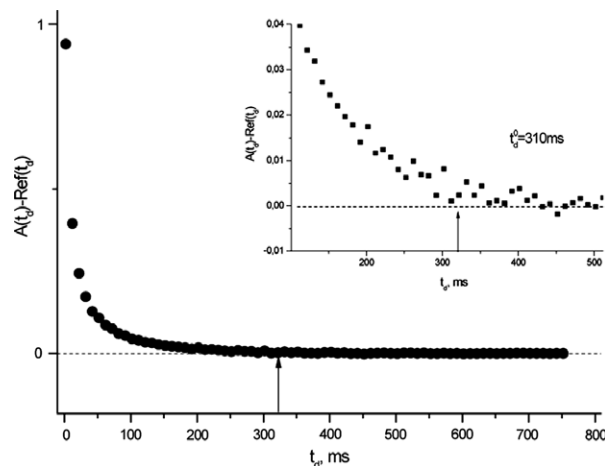


Fig. 7. $A(t_d) - \text{Ref}(t_d)$ for tridecane in partially (20%) filled quartz sand.

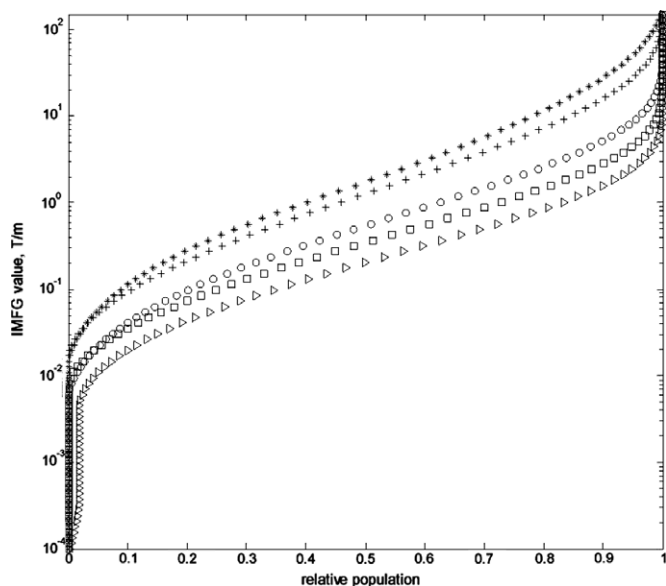


Fig. 8. IMFG distributions in the partially (20%) filled quartz sand at the diffusion times 5 ms (stars), 10 ms (pluses), 38 ms (circles), 80 ms (squares) and 157 ms (triangles).

fluid molecules are localized in the pore space within some “drops”, because in the case of its localization in the shape of thin film, diffusion path for the complete IMFG averaging should be of the order of magnitude of pore size. Thus, we can conclude that in the partially filled porous media distance of complete IMFG averaging is determined by the size of zones where diffusant molecules are localized.

Note that the studied porous media are characterized by the microporosity with the micropore size of about $5.6 \mu\text{m}$, thus, the question about fluid “drops” localization is still open.

3.2.3. Tau-scanning experiment

PFG NMR and DDif approaches allow us to conclude that fluid molecules are localized within some isolated zones (“drops”) in the porous space. Information about IMFG distribution and the features of its diffusion averaging was obtained by tau-scanning experiment. In Fig. 8, IMFG distributions are shown for the partially filled quartz sand in the diffusion time range from 5 to 157 ms. As one can see from the figure, apparent IMFG distributions are different at different diffusion times. The comparison of IMFG distributions for fully and partially filled porous space could be done by Figs. 5 and 8 comparison (the same symbols show distribution at the same diffusion times). As one can see from the figures, maximum IMFG values correspond to the minimum diffusion times for both partially and fully filled porous space. At the same time, speed of the diffusion averaging is different: as it can be seen from Fig. 8, diffusion time 5 ms corresponds to the second stage of the diffusion averaging process in the partially filled quartz sand. Findings indicate again that in the partially filled porous space

internal gradient averaging happens at the shorter distance in comparison with the same fully filled space. It is also interesting to note that even IMFG profiles are similar for the partially and fully filled porous media at the same diffusion times, the share of molecules in the areas with the high gradient values being different. Thus, detailed analysis indicates that the share of molecules in the areas with high gradients is bigger for the partially filled porous media. It says that in the partially filled quartz sand fluid molecules are mainly localized in the zones with high internal field gradients. It is well known that IMFG values grow up together with surface curvature, meaning that it is quite logical to assume that high IMFG value zones are localized in the pore granular contacts and in micropores.

4. Conclusions

Our study shows that introduced tau-scanning experiment allows us to obtain information both about IMFG profile in the porous media and the features of its diffusion averaging, at the same time DDif experiment gives numerical information about diffusion averaging but does not give information about averaging mechanisms. We demonstrate complex approach of porous media analysis by NMR. This approach includes three NMR techniques and allows also studying partially filled porous space and answering the question about fluid localization in it. In the studied system classical NMR and DDif data gave information about fluid localization within some isolated “drops” in the pore space. It was found also that drop average size corresponds to the scale of the IMFG diffusion averaging in the partially filled pore space. Note that this size (about $12 \mu\text{m}$) is sufficiently smaller than the pore size indicating that in the partially filled pore space diffusion averaging happens over the size of the zones where fluid is localized. Tau-scanning approach allowed one to obtain additional information about fluid localization in the pore space, namely, within fluid “drops” in the areas of granule contacts and microporosity.

We also note that different approach data are consistent and complementary that indicates perspectives of using introducing “triple” NMR approach to porous media study by NMR.

Acknowledgments

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