On the strong field dependence and nonlinear response to gadolinium contrast agent of proton transverse relaxation rates in dairy cream


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Abstract

Dairy cream, as a suspension of lipid droplets in water, is a potentially useful magnetic resonance imaging (MRI) phantom material and an interesting material for studying fundamental relaxation mechanisms. Here we report a strong increase in the transverse relaxation rates with field strength for both the water and lipid protons in dairy cream. Also, studies at 4.7 T reveal a nonlinear response of transverse relaxation rates with increasing concentration of a common gadolinium (Gd)-based contrast agent, including an initial decrease of water relaxation rates as measured with Hahn spin echoes at the lower Gd concentrations. The results are treated within the framework of a model in which the magnetic susceptibility difference between the lipid droplets and the aqueous phase plays the prominent role for transverse relaxation. Second-order polynomial fits of the water proton transverse relaxation rate dependence on field strength and on Gd concentration at 4.7 T provided experimental parameters from which model parameters are extracted and compared with expectations available from the literature.

Keywords: Dairy cream; Transverse relaxation; Magnetic resonance imaging (MRI); Phantom material; Field strength dependence; Gadolinium contrast agent

1. Introduction

Dairy cream has been suggested as a potentially useful phantom material for biexponential transverse relaxation time behavior [1] with particular relevance for assessing demanding constraints on Carr–Purcell–Meiboom–Gill (CPMG) imaging sequences [2] designed to extract the short T2 component from myelin-associated water [3,4]. More recently, dairy cream has been suggested as a phantom material for assessing diffusion imaging methods [5] designed to extract biexponential diffusion decay curves from tissue when measured over extended b-factor ranges [6,7]. The biexponential T2 and diffusion decays of dairy cream signal are attributed to the nonexchanging water and lipid proton pools, which also make dairy cream a suitable phantom material for assessing spectroscopic imaging techniques or fat suppression methods.

Because of its potential utility as an MR phantom material, it is of interest to characterize the relaxation properties of dairy cream protons. In this work, the field dependence of the transverse relaxation rates and the response of the transverse relaxation rates to a clinical gadolinium (Gd)-based contrast agent at 4.7 T have been measured. A strong field dependence of the water and lipid proton transverse relaxation rates was observed. The addition of the Gd contrast agent was observed to cause an anomalous decrease of water relaxation rates, as measured with Hahn spin echo methods, at low Gd concentrations. The finding is attributed to quenching one of the most important native transverse relaxation mechanisms in dairy cream—the microscopic magnetic field gradients at the lipid/water interface. The field dependence and dependence on Gd concentration at 4.7 T of the water transverse relaxation rate are analyzed using the simplest theoretical analysis available for transverse relaxation due to...
water diffusion within the gradients generated by weakly magnetized spheres [8–13].

2. Materials and methods

For data reported in this work, dairy cream (Heavy Cream, Ultra Pasteurized, Garelick Farms, Franklin, MA) was purchased prior to its expiration date and allowed to come to room temperature for scanning purposes. Scanning of the cream was performed between 4 and 8 h after purchase from the market-supplied refrigerated environment. This time window was found to allow for room temperature to be achieved while avoiding spoiling of the cream, which manifested as a separated, partially solidified fat layer observed in samples kept at room temperature overnight. Experimental measurements reported in this work resulted from separate measurements from a minimum of two and up to five different dairy cream lots except for the two lowest field measurements (0.2 and 0.5 T) where experiments were carried out only once on the same dairy cream phantom.

2.1. Transverse relaxation rate field dependence of dairy cream

The field dependence of the transverse relaxation rate of water and lipid protons in dairy cream was assessed by performing experiments at 0.2, 0.5, 1.5, 3.0, 4.7 and 8.5 T. For the 0.2- to 3-T field strengths, clinical scanners (General Electric Medical Systems, Milwaukee, WI) were used with standard quadrature head coils employed for transmission and reception. For the 4.7-T field strength, a horizontal bore Bruker Biospec (Bruker Instruments, Billerica, MA) was used with a volume coil for transmission and reception. For the 8.5-T measurements, a vertical bore Bruker scanner MSL 500 (Bruker Instruments, Billerica, MA) was employed with volume coil transmission and reception. At each field strength, a Hahn spin echo imaging sequence was used to acquire images at echo times (TEs) ranging from 20 to 300 ms with repetition times (TRs) of 1 s. For field strengths of 1.5 T and higher, receiver bandwidths were reduced to levels that permitted spatial separation of regions of pure water and pure lipid along the frequency encoding direction, permitting separate measurements of their dependence on TE. Only the bulk relaxation from the combined water and lipid proton signals was measured at 0.2 and 0.5 T due to the small chemical shift at these field strengths. Slice thickness and fields-of-view (FOVs) on the clinical scanners (3 T and below) were 10 and 240 mm, respectively. A 2-mm slice thickness and FOVs of 60 and 30 mm were used for the 4.7- and 8.5-T smaller bore scanners, respectively.

2.2. Relaxation rate dependence on Gd concentration

The commercially available contrast agent Gd-DTPA (Magnevist, Schering, Berlin, Germany) and dairy cream were mixed to make solutions with varying Gd concentrations from 0 to 3.2 mM. To test for any systematic or instrumental problems associated with the T2 measurements, water and acetone/water mixtures were also prepared of 75% water/25% acetone and 50% water/50% acetone (%‘s by volume) and doped with the same contrast agent to obtain solutions with Gd concentrations ranging from 0 to 3 mM. Experiments were performed at 4.7 T with a Bruker Biospec (Bruker Instrumentation, Billerica, MA). Samples were placed in a cylindrical phantom (diameter=3 cm, length=11 cm) and allowed to warm to room temperature (22±2°C) prior to experimentation. Temperature measurements were made before and after each scan to insue temperature stability to within 1°C for all measurements. During image acquisition, a low receiver bandwidth (10080 Hz) was set to allow a spatial separation of pure water and pure lipid components along the frequency encoding direction. To measure T1 relaxation times, spin echo images were acquired with a TE of 27 ms and 10 to 15 different TR values from 50 to 2500 ms with a 128×64 image matrix, 6×6 cm FOV, and a 2-mm slice thickness. Due to the relatively long T1 for pure water/acetone samples with no Gd, TR values from 700 to 3500 ms were sampled. To measure T2 relaxation times, a 16-echo CPMG imaging sequence was used with TR=5 s, TE=22 ms, 64×64 image matrix, 6×6 cm FOV and a 2-mm slice thickness. For the cream samples, T2 was also measured using a Hahn spin echo approach with a TR of 5 s, 32×64 image matrix, 6×6 cm FOV, a 2-mm slice thickness and eight TE values of 20, 25, 30, 35, 40, 45, 50 and 60 ms.

Relaxation rates were evaluated from the data assuming monoeponential relaxation and Levenberg–Marquardt nonlinear least square algorithms, as implemented in the Matlab software environment (The MathWorks, Natick, MA), for curve fitting. The Gd relaxivities were calculated from plots of relaxation rates vs. Gd concentration. Linear regression was used to evaluate water and acetone transverse and longitudinal relaxivities according to $1/T_{1,2} = A + Bx$, where $x$ is the millimolar concentration of Gd. Linear regression was also used to calculate the longitudinal Gd relaxivities of water and lipid dairy cream protons. For the dairy cream transverse relaxation rates vs. Gd concentrations, however, it was necessary to add a quadratic term so that water and lipid transverse relaxation rates vs. Gd were fit with a second-order polynomial equation of the form $R_2 = 1/T_2 = A + Bx + Cx^2$.

3. Results

Fig. 1 is a plot of the means and standard deviations of dairy cream lipid and water transverse relaxation rates as a function of field strength $B_m$. A quadratic fit to the water proton data (including the lipid+water points at the two lowest field strengths), $R_2 = R_{2o} + \eta B_m^2$, is shown as the solid
line in Fig. 1 with $R_2$ and $\eta$ values being 9.6 s$^{-1}$ and 0.81 s$^{-1}$ T$^{-1}$, respectively.

Fig. 2 presents transverse relaxation rates of water and lipid dairy cream protons vs. Gd concentration. Results from both the Hahn spin echo experiments and the CPMG experiments are shown. The relaxation rates for the Hahn spin echo experiments are higher than those from the CPMG experiments, although the general trends for the transverse relaxation rates vs. Gd concentration curves are similar and clearly do not show the standard linear increase of relaxation rate with Gd concentration. These curves are amenable to fits with second-order polynomial functions shown as lines through the data. Table 1 provides the coefficients $A$, $B$ and $C$ found from these fits whose correlation coefficients were above 0.94.

Fig. 3 presents the longitudinal relaxation rates vs. Gd concentration for water and lipid protons in dairy cream and the linear regression fits to the data (solid lines). The slope of the water relaxation rate vs. Gd concentration is 5.29 s$^{-1}$ mM$^{-1}$ with a correlation coefficient of 0.99. The lipid proton longitudinal relaxation rate is virtually unaffected by Gd with a slope of 0.01 s$^{-1}$ mM$^{-1}$ and correlation coefficient of 0.07.

Fig. 4 presents the longitudinal and transverse relaxation rates of water and acetone protons as a function of Gd-DTPA concentration for the 75%-water/25%-acetone mixtures with their corresponding linear regression analyses. Similar plots were generated for the doped water

<table>
<thead>
<tr>
<th>Coefficients of the second-order polynomial fits of the transverse relaxation rates vs. Gd for dairy cream protons shown in Fig. 2</th>
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<tbody>
<tr>
<td>Water (Hahn)</td>
</tr>
<tr>
<td>$A$ (s$^{-1}$)</td>
</tr>
<tr>
<td>30.1</td>
</tr>
<tr>
<td>50.1</td>
</tr>
<tr>
<td>21.0</td>
</tr>
<tr>
<td>25.0</td>
</tr>
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</table>
solutions and the doped 50%-water/50%-acetone solutions. Table 2 provides the water Gd relaxivities, $r_{1w}$ and $r_{2w}$, and the acetone Gd relaxivities, $r_{1a}$ and $r_{2a}$, obtained from the slopes of the relaxation rates vs. Gd-DTPA concentrations for the three mixtures studied. The regression coefficients of the linear fits used to obtain the Gd relaxivities were above 0.98. Notice that as the volume fraction of acetone increases from 0% to 50%, the longitudinal and transverse Gd relaxivities of the water protons increase, whereas the Gd relaxivities of the acetone protons remain constant.

Fig. 3. Longitudinal relaxation rates vs. Gd concentration for water protons and lipid protons in dairy cream as measured at 4.7 T along with linear regression fits to the data. The lipid proton longitudinal relaxation is virtually unaffected by the Gd, while the Gd relaxivity of the water protons as measured from the slope of the linear regression fit was 5.29 s$^{-1}$ mM$^{-1}$.

Fig. 4. Transverse and longitudinal relaxation rates of water and acetone protons as a function of Gd-DTPA concentration in the 75% water/25% acetone solutions as measured at 4.7 T along with linear regression fits from which Gd relaxivities were calculated (Table 2).
protons do not vary substantially between the 25%- and 50%-acetone solutions.

### 4. Discussion

Before discussing the findings in dairy cream, the rationale for performing the Gd-doping experiments in the water/acetone solutions and the relevance of those experiments to the dairy cream results are addressed. The acetone/water-doping experiments were performed primarily to demonstrate a lack of any systematic or instrumental problems associated with our T2 measurements. When the first few experiments in dairy cream revealed nonlinear relaxation rate vs. Gd concentration behavior (Fig. 2) and even a decrease in the Hahn echo-based water transverse relaxation rates with increasing Gd, it was considered prudent to perform control experiments in water and water/acetone mixtures. The latter solutions were selected as they provided a pool of protons chemically shifted from water to roughly the same degree as the lipid protons in dairy cream. The control experiments revealed the anticipated linear dependence of the longitudinal and transverse relaxation rates on Gd concentration for both the water and the acetone protons (Fig. 4), ruling out any significant systematic or instrumental inaccuracies with the methodology. The Gd relaxivities of the water protons were generally higher than that of the acetone protons (Table 2) and, unlike acetone protons, were found to increase as the water concentration in the solution decreased. It is suggested that acetone relaxation is dominated by outer sphere relaxation effects and so is largely independent of the acetone concentration. Water molecules do have access to the first coordination sphere of the Gd ion, and so, for a fixed Gd concentration, reducing the water concentration leads to a higher probability of first coordination sphere interactions, increasing the relaxivity roughly in inverse proportion to the water concentration. Since Gd-DTPA-doped acetone/water solutions can be used for calibrating spectroscopic techniques designed to quantify water and fat [11,12], knowledge of the proton Gd relaxivities as provided in Table 2 for a 4.7-T field strength is of general utility. Finally, the value for the transverse relaxation rate of pure water at 4.7 T measured from these experiments was incorporated into a specific model for dairy cream water proton transverse relaxation, as now discussed.

The transverse relaxation rates of both the water and lipid protons within dairy cream show a pronounced dependence on field strength (Fig. 1) and an interesting, nonlinear dependence on Gd concentration (Fig. 2). We attribute this behavior to microscopic field gradients at the lipid droplet interface associated with the magnetic susceptibility difference between the lipid and aqueous phases. The following discussion focuses primarily on an analysis of the water transverse relaxation rates as measured from the Hahn spin echo experiments for which theoretical treatments are available [8–13].

It is assumed that the primary dephasing mechanism for water is from diffusion within microscopic field gradients as the water molecules pass through the “outer sphere” regions of the droplets. In the so-called motionally narrowed limit, it is assumed that rapid spatial diffusion of water protons allows them to experience the full range of frequencies available from the microscopic field gradients in a short period of time. This condition is characterized by the criterion \( R_L^2/D \delta\omega < 1 \), where \( R_L \) is the radius of the lipid droplet, \( D \) the diffusion coefficient of water and \( \delta\omega \) the spread of Larmor frequencies characterizing the microscopic field gradients near the surface of the lipid droplets [12].

Under these conditions, the relaxation rate of water protons from Hahn spin echo sequences may be approximated with an equation derived by Gillis and Koenig [11] for solvent relaxivity due to particulate susceptibility variations and employed previously by Marciani et al. [12] in analyzing transverse relaxation rates in fat emulsifications. The basic equation governing the water transverse relaxation rate \( R_{2w} \) under these conditions is

\[
R_{2w} = R_{2wo} + \alpha(\lambda_L - \lambda_w)^2,
\]

where the constant \( \alpha \) is given by the formula:

\[
\alpha = (16/135)f(\gamma B_o/3)R_L^2/D
\]

and \( R_{2wo} \) is the intrinsic water relaxation rate; \( f \) is the fraction of the particulate, in this case the lipid volume fraction; \( \gamma \) is the proton gyromagnetic ratio \( (2\pi 4258 \text{ Hz Gauss}^{-1}) \); \( B_o \) is the field strength; and \( \lambda_L \) and \( \lambda_w \) are the magnetic susceptibilities of the lipid and water phases, respectively [11,12].

For the case of Gd addition to the water phase, we add to this Eq. (1) a linear contribution to the relaxation from the Gd paramagnetic agent as well as a linear increase in the magnetic susceptibility of the aqueous phase with Gd concentration. From these considerations, the following equation for the transverse relaxation of the water protons \( R_{2w} \) in dairy cream is postulated:

\[
R_{2w} = R_{2wo} + R + \alpha(\lambda_L - \lambda_w)^2
= R_{2wo} + R + \alpha(\lambda_L - \lambda_{w0} - \beta x)^2
\]

and \( R \) is the transverse water Gd relaxivity in per second per millimolar. The term in parentheses represents the difference between the lipid magnetic susceptibility \( \lambda_L \) and the water magnetic susceptibility \( \lambda_w \), which is taken to be \( \lambda_{w0} + \beta x \), with \( \lambda_{w0} \) being the water magnetic susceptibility in the

### Table 2

Longitudinal and transverse Gd relaxivities of water and acetone protons in the three solutions studied

<table>
<thead>
<tr>
<th></th>
<th>( r_{1w} ) (s(^{-1}))</th>
<th>( r_{1a} ) (s(^{-1}))</th>
<th>( r_{2w} ) (s(^{-1}))</th>
<th>( r_{2a} ) (s(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>3.26</td>
<td>5.62</td>
<td></td>
<td></td>
</tr>
<tr>
<td>75% water/25% acetone</td>
<td>4.90</td>
<td>1.73</td>
<td>9.47</td>
<td>2.78</td>
</tr>
<tr>
<td>50% water/50% acetone</td>
<td>6.86</td>
<td>1.68</td>
<td>15.39</td>
<td>2.65</td>
</tr>
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</table>
absence of Gd and $\beta$ being a proportionality constant representing a linear increase of water susceptibility with Gd concentration. The variation of the water magnetic susceptibility with contrast agent, without an accompanying change in the lipid magnetic susceptibility, assumes that the Gd-DTPA is dispersed solely within the water phase. This assumption is consistent with the lack of any effect of Gd concentration on the lipid longitudinal relaxation rate, as shown in Fig. 3.

We first consider the field dependence of the water proton transverse relaxivity in the absence of Gd and use Eq. (1) to link the quadratic coefficient $\eta$, measured to be 0.81 s$^{-1}$ T$^{-2}$ from the fit to the experimental data in Fig. 1, to the model parameters through the relation

$$\eta = (16/135)f(\gamma/3)^2(\lambda_L - \lambda_{wo})^2R_L^2/D$$

(4)

The room temperature diffusion coefficient of water in heavy-cream samples has been previously measured to be approximately 1.4×10$^{-9}$ m$^2$s$^{-1}$ [5]. Estimates of the magnetic susceptibility values for lipid and water are extracted from the articles by Szczepaniak et al. [15] and Hopkins and Wehrli [16]. From these sources, the lipid and water magnetic susceptibilities, in MKS units, are taken to be $-8.44$ and $-9.05$ ppm, respectively, so that their difference ($\lambda_L - \lambda_{wo}$) is approximately 0.61×10$^{-6}$. The volume fraction ($f$) of lipid in our cream samples is estimated from the milk fat % information available from the product label as 0.38. Substitution of these values into Eq. (1) in combination with the measured value of $\eta$ leads to an estimate for the lipid droplet radius ($R_L$) of 2.9 $\mu$m. The spherical droplets in cream, referred to as milk fat globules in the dairy literature [17,18], are reported to have radii ranging from 0.1 to 8 $\mu$m with mean radii reported to be approximately 2 $\mu$m, in satisfactory agreement with our analysis considering the number of approximations involved.

Eq. (3) is now applied to analyze the Gd-doping experiments. Upon inspection, the following relationships between the coefficients of the empirically obtained second-order polynomial fit $A$, $B$ and $C$, and model parameters are obtained:

$$A = \alpha(\lambda_L - \lambda_{wo})^2 + R_{2wo}$$

(5a)

$$B = R - 2\alpha(\lambda_L - \lambda_{wo})\beta$$

(5b)

$$C = 2\beta^2$$

(5c)

Technically, these equations represent a system of five unknowns on the right and three knowns on the left. Again, we utilize an independent estimate of the magnetic susceptibilities [15,16] and further assume that the relaxation rate of pure water, $R_{2wo}$, is equal to that measured from our experiments in pure water at 4.7 T, $R_{2wo}$=2.4 s$^{-1}$. Using the values of $A$, $B$ and $C$ in Table 1 for water protons of the Hahn spin echo experiment, the remaining unknown parameters $\alpha$, $\beta$ and $R$ can be calculated. Using the $A$ value of 30.1 s$^{-1}$, Eq. (5a) provides an $\alpha$ value of 7.4×10$^{13}$ s$^{-1}$. Using this value of $\alpha$ along with Eq. (2) and the same values for the lipid volume fraction $f$ and water diffusion coefficient $D$, an $R_L$ value of 3.6 $\mu$m is obtained. This is in reasonable agreement with literature values for milk fat globule radii and similar to the value of 2.9 $\mu$m obtained above from the field dependence studies. Substituting the numerical value of $\alpha$ just calculated into Eq. (5c) and using the measured value of $C$ of 6.7 s$^{-1}$ m$^{-2}$ from Table 1, a value for $\beta$ of 0.30 ppm m$^{-1}$ is obtained. The $\beta$ parameter is the coefficient of linear increase of water magnetic susceptibility with Gd concentration and has been reported to be, in centimeter–gram–second unit, on the order of 0.026 cm$^3$mol$^{-1}$=0.026 ppm m$^{-1}$ [19–21]. Multiplying this value by 4$\pi$ to convert to MKS units leads to a value of 0.33 ppm m$^{-1}$, in close agreement with our $\beta$ value of 0.30 ppm m$^{-1}$. Finally, inserting the numerical values of $\alpha$ and $\beta$ just obtained into Eq. (5b) and using the measured value for $B$ of $-7.7$ s$^{-1}$ m$^{-2}$, an estimate for $R$ of 19.5 s$^{-1}$ mm$^{-1}$ is obtained. The transverse Gd relaxivity $R$ that we calculate from this analysis, 19.5 s$^{-1}$ mm$^{-1}$, is nearly four times higher than the transverse Gd relaxivity that we measured in pure water (Table 2). This is not unanticipated as Stanisz and Henkelman [22] have shown how the presence of macromolecular substituents, including those found in powdered milk, can dramatically increase the Gd relaxivities compared to values measured in pure water or saline, an effect attributed to changes of the relevant correlation times of the Gd-DTPA and/or water molecules. Indeed, we detected a higher Gd longitudinal relaxivity for the water protons in dairy cream as opposed to pure water. Thus, the higher water proton transverse Gd relaxivity we extract in dairy cream is attributed to changes in the relevant correlation times affecting the water/Gd-DTPA relaxation interactions as mediated by the lipid and macromolecular constituents.

Although we have focused on an analysis of the water proton transverse relaxation as measured with the Hahn spin echo experiments, the same set of experiments also showed that the lipid proton transverse relaxation showed a strong field dependence (Fig. 1) and an interesting, nonlinear dependence on Gd concentration, which was reasonably well modeled with the second-order polynomial approach (Fig. 2 and Table 1). Both these observations suggest that the microscopic magnetic field gradients formed by the magnetic susceptibility difference between the lipid and water phases play a major role in the transverse relaxation of the lipid protons, although no theoretical analyses of this effect are, to our knowledge, available.

Finally, we found major differences in the transverse relaxation rates of both water and lipid protons when measured using Hahn vs. CPMG spin echo approaches with the latter sequence resulting in substantially lower relaxation rates (Fig. 2). It is well known that repeated refocusing 180 pulses restore transverse magnetization when diffusion within field gradients is an important contributor to relaxa-
tion. This effect most certainly accounts for the lower water relaxation rates obtained by CPMG vs. Hahn spin echo, and there is probably a wealth of information available from echo spacing dependence studies of water protons in dairy cream at high field, a subject beyond the scope of the present work. It is difficult to understand how diffusion within the local field gradients can play much of a role for the lipid protons due to the low diffusion coefficients of the larger lipid molecules [5]. There is, however, still a substantially smaller due to the low diffusion coefficients of the larger lipid field gradients can play much of a role for the lipid protons

It is difficult to understand how diffusion within the local

5. Conclusions

This is the first report, to our knowledge, in which the addition of a paramagnetic agent to a substance has resulted in a decrease in $^1$H transverse relaxation rates as opposed to an increase. Although seemingly anomalous, the finding is quite consistent with current models of water T2 relaxation from susceptibility-induced gradients and the distribution of typical Gd contrast material. There have been other studies in which the magnetic susceptibility of one phase of two-phase materials has been paramagnetically doped in order to alter relaxation and/or susceptibility properties [11,13,16]. The lower field strengths used in those studies most probably precluded a direct observation of a decrease in the transverse relaxation rate as this requires the $B$ coefficient in the polynomial fit of the Gd relaxivity curve to be negative, and this is more likely as field strength increases [see Eq. (5b)] due to the quadratic dependence of $x$ on $B_o$. Regardless, it is of interest to speculate on possible clinical ramifications where the effect we observe may play a role. For example, red marrow and fatty liver conditions, found in toxic liver injury or metabolic disorders, provide MRI voxels with lipid and water protons, and magnetic susceptibility differences between the two phases most probably contribute to the observed transverse relaxation. Thus, the introduction of Gd-DTPA or other paramagnetic agents may not result in a direct linear increase of transverse relaxations. The effect should become more pronounced at the higher field strengths, such as 3 T, now becoming more routinely available for clinical use.

To conclude, dairy cream, which has previously been suggested as a potential phantom material for biexponential T2 [1] and diffusion [5] studies, also appears to be an interesting material for studying the fundamental T2 relaxation mechanisms associated with magnetic susceptibility differences.

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References


