Towards Low-Cost Magnetic Resonance Relaxometry †

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† Presented at the 12th International Electronic Conference on Sensors and Applications (ECSA-12), 12–14 November 2025; Available online: https://sciforum.net/event/ECSA-12.

Abstract

Magnetic Resonance Relaxometry is a powerful technique which reveals a sample's molecular dynamics thanks to the dependence of the T1 relaxation time on field strength. With applications in protein research, food systems, materials development and environmental science, relaxometry measurements are typically undertaken using a technique known as fast field cycling where T1 is measured a given detection field, but the sample experiences relaxation in a variable field without the challenges associated with retuning a probe to each of the necessary frequencies of interest. This technique is limited by a maximum relaxation time since the measurement and relaxation fields are typically applied using a fluid cooled electromagnet which ultimately will overheat for very long experimental times. In this work we propose an alternative approach to permit measurements of samples with inherently long T1. We utilise a broadband spectrometer alongside a solenoid transmit-receive coil and custom tuning and matching boards whilst two sets of magnets are moved around the coil to achieve a range of different fields. By collecting a reduced number of points and utilising this method, we show it is still possible to make useful measurements on samples at a range of frequencies which has great potential in quality assurance applications. We find a similar trend for food samples of corn oil while Manganese Chloride, a common contrast agent, has a more than 100% difference when compared to traditional fast field cycling measurements.

Keywords: magnetic resonance relaxometry; fast field-cycling; low cost; benchtop

1. Introduction

Spin-lattice, or longitudinal, relaxation is the process of a nuclear system's net magnetisation (M) returning to its initial maximum value (M_0) following excitation by application of radio frequency energy [1]. Energy is transferred to neighbouring molecules, the magnitude of which is given by Equation (1) [2]:

$$M_z = M_0 \left(1 - e^{-\frac{\tau}{T_1}}\right) \tag{1}$$

where M_z is the net magnetisation along the z axis, M_0 is the initial magnetisation, τ is the measurement time and T_1 is a sample property characterisation this exchange of energy. Measuring T_1 reveals interesting sample properties such as molecular viscosity and protein morphology. To do so, several data points must be acquired to which an exponential with time constant T_1 is fitted. Such an experiment is the inversion recovery experiment

where the magnetisation is initially inverted by applying a radiofrequency (RF) pulse of 180° before allowing the system to relax for a time (τ). Following this delay, a 90° RF pulse is applied, and the resulting free induction decay (FID) is Fourier transformed to give a spectrum. The intensity of the spectral peaks are then plotted against the varying τ values [3]. The value of T_1 is defined as the time taken for M_z to return to ~63% of its original value. It typically takes a spin system $5*T_1$ to recover 99% of its original equilibrium magnetisation.

T_1 is also dependent on the magnetic field strength. With interesting features encoded in the relationship between T_1 and field strength. It is for example possible to measure the coupling of protons to nitrogen atoms by measuring T_1 at the field strengths where the nitrogen quadrupolar coupling coincides with the proton frequency [4]. Measurement of the variation of T_1 with field strength is commonly achieved using fast field cycling (FFC) experiments. An FFC relaxometry experiment consists of a repeated T_1 measurement pulse sequence, such as an inversion recovery sequence.
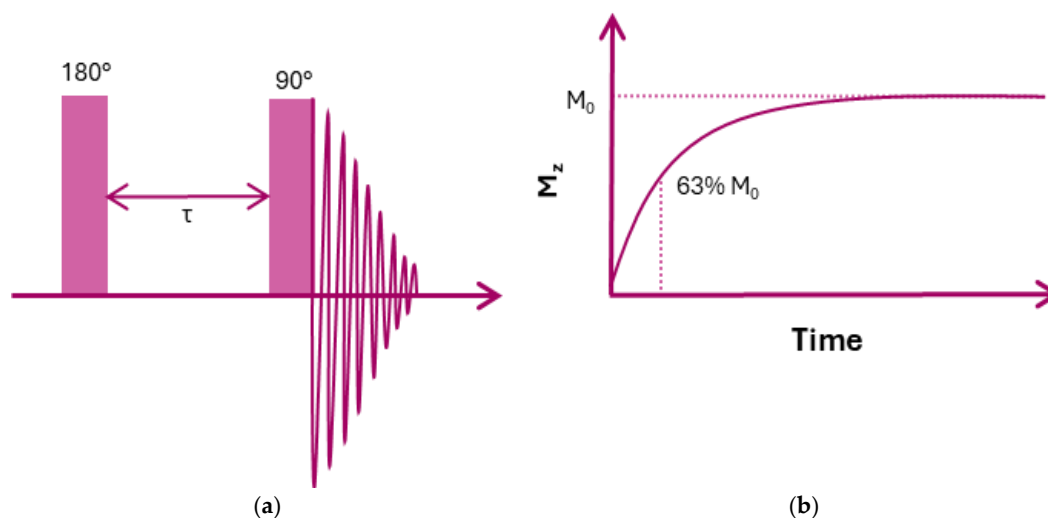

(**a**) (**b**)

Figure 1. (**a**) Pulse sequence diagram for an inversion recovery experiment and (**b**) model of the longitudinal relaxation process.

The excitation occurs at the same field strength and the FID is detected at the same detection field for each repetition; however, a relaxation field (and therefore the Larmor frequency) is varied. A graph of the relaxation rate ($1/T_1$) against the range of Larmor frequencies can then be produced.

Typical field cycling experiments will consist of either a pre-polarised (PP) or non-polarised (NP) sequence. During a PP sequence, the sample will first build up an equilibrium magnetisation in a polarisation field for a period d_p, before switching to a lower relaxation field where the spin system relaxes to a new equilibrium magnetisation value. It will then switch to a detection field where the 90° RF pulse is applied, and the resulting FID detected. The time needed to switch between these fields is defined as the switching time s_w. During a NP sequence, there is no polarisation field, and the equilibrium magnetisation is instead built up in the relaxation field. PP sequences allow for the exploration of much lower fields, whilst NP sequences are used to bridge the gap between these fields and the fields at which more conventional techniques can be used [5].
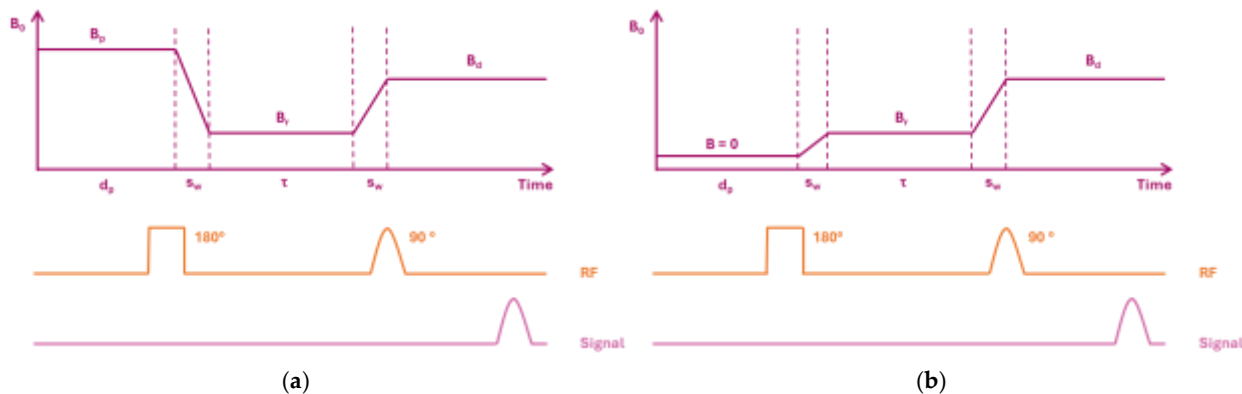
Figure 2. (**a**) PP sequence and (**b**) NP sequence.

One of the main drawbacks of FFC systems is that they require robust cooling systems and are expensive to purchase. Furthermore, to prevent overheating of the magnetic field coils, they are restricted to exploration of samples with relatively short T_1 limiting their applicability to low concentrations of aqueous samples. A block diagram of a typical FFC system can be seen in Figure 3 [3]. It is the goal within this work to produce a lower cost system that utilises permanent magnets that can be moved relative to the sample to give varying field strengths and use this setup to show comparable results with an existing FFC system across a select range of Larmor frequencies.
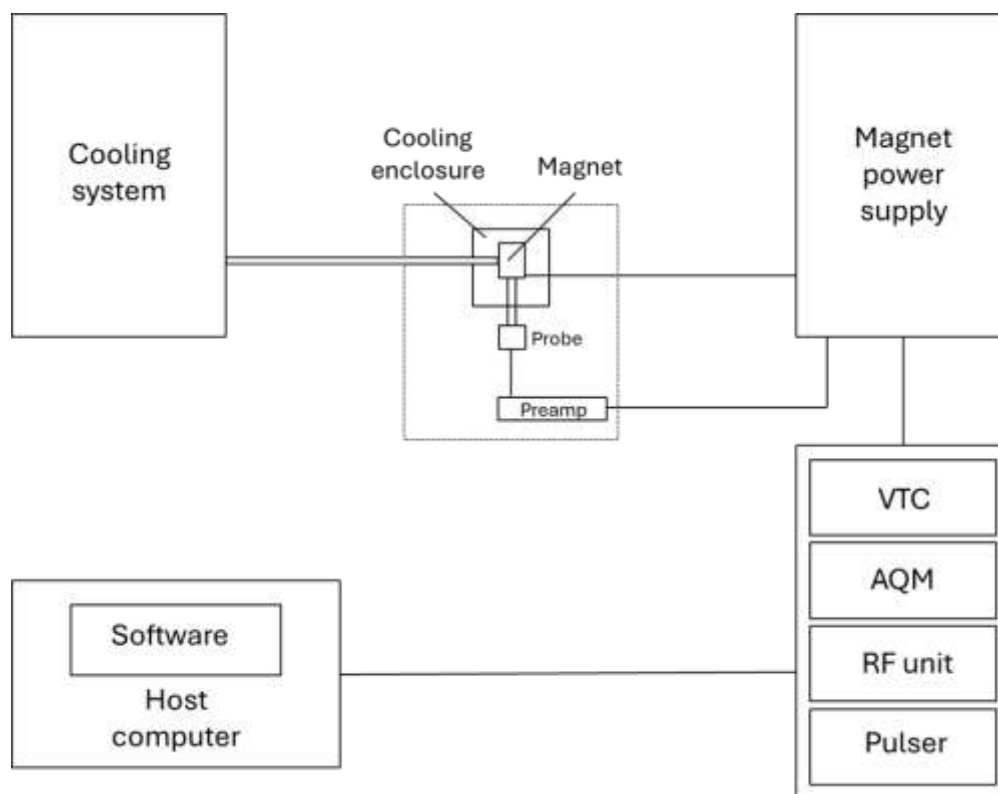

Figure 3. FFC instrument with variable temperature controller (VTC) and acquisition module (AQM).

2. Materials and Methods

Two pairs of 50 × 50 × 10 mm N45 Neodymium magnets (Magnosphere, Germany) were assembled in acrylic holders and mounted onto lengths of M10 threaded rod. These were secured in place using a set of 16 nuts, one either side of the holders at both ends. Steel yokes measuring 100 × 40 × 10 mm were added to the outer faces of the magnets, with an additional 250 × 40 × 10 mm length underneath to produce a more stable magnetic field in the central region for measurements by providing a return path for the stray magnetic field. This setup can be seen below in Figure 4.

Figure 4. Photograph of variable field sensor showing variable position magnet housings and suspended RF coil and sample.

The spacing between the two sets of magnets is currently manually adjusted by moving the nuts. This will be automatable by using reverse thread nuts on one acrylic holder and stepper motor geared to the threaded rods. The resulting field was then measured to determine the tuning frequency for the coil using a gauss meter (HM08 Hirst Magnetics, Falmouth, UK). With this setup, it is possible to achieve a maximum field strength of up to 400 mT.

A single layer 13-turn copper solenoid coil was tuned to each individual measurement field using a custom tuning and matching board, from a range of 300 mT (12.7 MHz) down to 200 mT (8.5 MHz). Shielding was also placed around both the coil and the tuning and matching board to improve the signal-to-noise ratio (SNR) in electrically noisy environments. The sample was positioned within the field using a retort stand and clamp.

Using a benchtop NMR console (Kea^2, Magritek, New Zealand) a series of T_1 inversion recovery experiments were run for two samples. Each scan consisted of 64 averages across a τ range of 1 ms-1000 ms for a sample of corn oil, and a range of 1 ms-150 ms for a sample of $MnCl_2$.

Identical samples were also analysed using a FFC NMR relaxometer (SPINMASTER, Stelar, Italy) across a frequency range of 15 MHz to 5 MHz using a NP sequence with 8 averages. The samples were kept at room temperature (293 K) for the duration of the scans.

3. Results and Discussion

Figure 5 shows a comparison between the results obtained using the FFC relaxometer and the variable field sensor for both corn oil and $MnCl_2$. The relaxation rate for both samples obtained using this system follows the expected trend with increasing Larmor frequency, however, as can be seen there is a significant difference between these values and those obtained using the FFC relaxometer which is more pronounced for the manganese chloride samples.
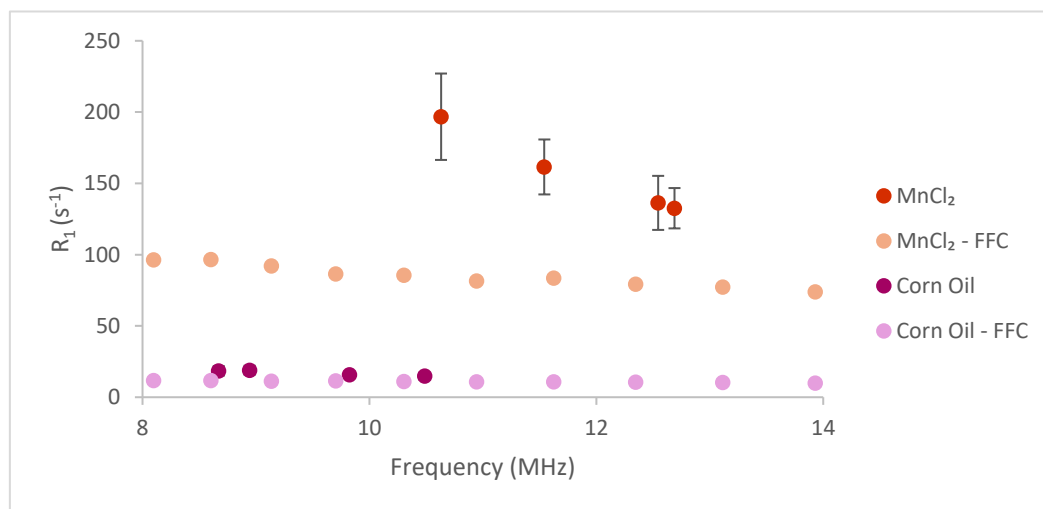

Figure 5. Plot of resulting relaxation rates against frequency for samples of $MnCl_2$ and corn oil using both the new magnet system and the FFC relaxometer.

Errors in the results obtained by the new system for both samples are also notably larger than for the FFC relaxometer. There are several potential contributing factors to this, the most prominent likely being that the field within the centre of the magnets is not as stable as it would ideally be for these measurements, and therefore it is likely that the sample is sitting over a range of different field strengths. This could be rectified in the future by improving the design of the yokes for a more stable central field or the inclusion of shaped pole pieces. A more accurate method for positioning the sample within the field as well as improved shielding for both the sample and the tuning and matching board would also help with reducing the noise and the errors in the measurements.

These preliminary results require further investigation to using a greater range of concentrations to better understand the source of the discrepancy.

4. Conclusions

We have presented preliminary results demonstrating the ability of an automatable permanent magnet sensor to create a T_1 frequency dispersion plot akin to that of a fast field cycling system. There is however a significant discrepancy between the results of a traditional fast field cycler and our new sensor which requires further investigation. This will include measurement of different paramagnetic agents at different concentrations to determine if the offset is consistent or variable.

Further development is needed to improve this system, including enhancing the stability of the field in the centre of the magnets with better positioned and designed yokes. Improvements to the SNR could also be made by increasing the number of averages per scan and further tweaks to the shielding around the coil and tuning and matching board. It would also be beneficial to investigate a wider range of frequencies to better test and understand the limits of the system. Once this optimisation is concluded, the system can be automated to provide automatic measurements of T_1 dispersion.

Author Contributions:

Funding:

Institutional Review Board Statement:

Informed Consent Statement:

Data Availability Statement:

Conflicts of Interest:

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