Development and Applications of a Low-Field Portable NMR System

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Abstract

Nuclear magnetic resonance (NMR) is a phenomenon similar to MRI in which radio frequency signals are used to excite and manipulate atomic nuclei within a static magnetic field. Following excitation, the nuclei return to equilibrium, all the while offering valuable molecular level information pertaining to the sample.

Within the last decade, the development of small and inexpensive NMR spectrometers and permanent magnet NMR sensors has been a significant focus within the NMR community. More recently, application scientists have sought practical applications for the new technologies.

In this thesis, a prototype NMR apparatus consisting of a spectrometer and 3.2MHz permanent magnet sensor was extended to enable scientific measurements. This involved developing radio frequency electronic circuitry for the spectrometer front-end, and electromagnetic noise shielding and temperature regulation for the magnetic sensor. Experimental results confirmed that repeatable measurements using the modified apparatus were indeed possible.

The NMR apparatus was thereafter successfully used to study flow, diffusion and kiwifruit using several different experimental techniques. A significantly larger effort was then expended upon the study of T_2 relaxation in pectin model systems using pH as the adjustable parameter. The fascinating experimental results were successfully interpreted and modeled across three pH zones in terms of a proton chemical exchange model and molecular conformational changes. In addition, it was found that pectin carboxyl de-protonation was significantly less than expected. Further experiments performed upon galacturonic acid monomers, dimers and trimers appeared to further illuminate the pectin results. Future experiments are planned.

Also while studying pectin solutions, an unexpected pH-dependent water transverse relaxation behavior was observed at both 3.2MHz and 400MHz. The only references found in the literature were from a small publication almost 50 years ago, and a 2011 publication.

Altogether, this thesis contributed to original knowledge in several ways: it showed how a low-field apparatus and single-sided sensor could be improved and utilized for a variety of scientific measurements; it showed both experimentally and theoretically how T_2 for pectin solutions change with pH; it revealed an unexpected de-protonation limit for pectin molecules; it revealed a T_2 pH dependence for water.

To my wife Liz
who has endured
much over the years.

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List of abbreviations

γ	The magnetogyric (or gyromagnetic) ratio
ω	Frequency in radians per second
ω_{eff}	Effective frequency in the rotating frame in radians per second
A	Amps
AFM	Atomic force microscopy
AC	Alternating current
ARRL	American Radio Relay League
B_0	Static magnetic field oriented along the z-axis
B_1	Oscillating magnetic field oriented along the x-axis
CE	capillary electrophoresis
CC	Counterion condensation
CLI	Command line interface
COOH	Carboxyl
CPL	Coupled
CP	Carr Purcell
CPMG	Carr Purcell Meiboom Gill
CSV	Comma separated values
CTR	Current transfer ratio
D-D	Dipole-dipole coupling
Da	Dalton: $1/12^{\text{th}}$ of the mass of a carbon atom at rest in its ground state
DC	Direct current
DE	Direct echo
DM	Degree of methyl-esterification
DP	Degree of polymerization
DSP	Digital signal processor
EMF	Electro motive force, or voltage
EMI	Electromagnetic interference
FID	Free induction decay
GalA	Galacturonic acid
GDL	Glucono delta-lactone
GUI	Graphical user interface

HCl	Hydrochloric acid
HDD	Hard disk drive
HM	High methoxyl
HPA	High-power amplifier
Hz	Frequency in Hertz
IC	Integrated circuit
$J(\omega)$	Spectral density as a function of frequency
kDa	Kilodalton
Kea	NMR spectrometer
kHz	Kilohertz
LED	Light emitting diode
LM	Low methoxyl
M	Molarity, measured in $\text{mol}\cdot\text{L}^{-1}$
M_r	Molecular weight
μA	Micro amps
μF	Micro Farads
μH	Micro Henries
mA	Milli amps
Mole	MObile Lateral Explorer or MObile Liquid Explorer
mole	$\sim 6.022 \times 10^{23}$ atoms of molecules
MRI	Magnetic resonance imaging
nF	Nano Farads
NaOH	Sodium hydroxide
NdFeB	Neodymium iron boron
NIB	Neodymium iron boron
NMR	Nuclear magnetic resonance
OH	Hydroxyl
pK_a	Acid dissociation constant
PAD	Pre-amplifier duplexer
PCB	Printed circuit board
(π)	180° RF pulse
$(\pi/2)$	90° RF pulse
pF	Pico Farads
PID	Proportional-integral-derivative
Prospa	NMR software
PSU	Power supply unit
Pt100	Platinum resistance thermometer (100Ω resistance at 0°C)
QST	Amateur radio enthusiasts magazine published by the ARRL
Q	Quality factor of a inductor, capacitor, or tuned circuit
R_1	Longitudinal (or spin-lattice) relaxation rate

R_2	Transverse (or spin-spin) relaxation rate
RX	Receive or receiver
RF	Radio frequency
RMS	Root mean square
R_{eff}	Concentration ratio of calcium ions to de-protonated carboxyls
RS	Radio Spares Components
RS-485	Serial differential hardware communications protocol
SAR	Specific absorption rate
SAXS	Small-angle X-ray scattering
SmCo	Samarium Cobalt
SMPSU	Switched mode power supply unit
SNR	Signal to noise ratio
SE	Stimulated echo
SMA	SubMiniature version A
SMB	SubMiniature version B
T	Magnetic flux density (Tesla)
T_1	Longitudinal (or spin-lattice) relaxation time
$T_{1\rho}$	Longitudinal (or spin-lattice) relaxation time in the rotating frame
T_2	Transverse (or spin-spin) relaxation time
t_E	Echo time
TX	Transmit or transmitter
USB	Universal serial bus
V	Volts
wt%	Weight percent

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