

Non-destructive evaluation of concrete and wood properties using NMR

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In this paper, a preliminary investigation of the Nuclear Magnetic Resonance (NMR) technique for non-destructive evaluation of concrete and wood is made. Basic principles of NMR are discussed. Samples of concrete and wood were tested using NMR. In these tests, the hydration process in concrete and the moisture content of wood samples were monitored. In addition, standard destructive testing of these materials was conducted to determine their failure strengths. The results of destructive tests were compared with those of NMR testings. Good correlation was observed between the results of non-destructive NMR and standard destructive tests.

Introduction

Accurate determination of the *in situ* physical properties of construction materials has been a primary objective of many researchers and engineers. For this purpose, many non-destructive techniques with various degrees of accuracy have been developed. In this paper, the potential of Nuclear Magnetic Resonance (NMR) as a possible tool for non-destructive measurement of strength of concrete and wood will be explored. NMR is particularly suited for measurement of chemically-bound hydrogen, and free hydrogen in the form of water and other exchangeable hydrogens in concrete and wood samples. Therefore, this method possesses a good potential for monitoring the hydration process in concrete and its relationship to the concrete compressive strength, as well as for determining the effect of moisture content on the strength of wood.

NMR has been extensively used in a variety of disciplines such as chemistry, biology and medicine. However, its application as a non-destructive test method for construction materials has been very limited. It is primarily used for studies of organic materials. Presently, NMR is one of the most powerful material analysis methods in biochemistry and analytical chemistry. In medicine, imaging by NMR is becoming increasingly popular as an effective diagnostic tool in recent years.

In this paper, a brief summary of the principles involved in NMR will be given. Preliminary results of non-destructive tests performed on concrete samples with different water/cement ratios and wood samples with different moisture contents will be discussed. The non-destructive test results will also be compared with a series of destructive tests performed on samples similar to those tested with NMR to investigate any correlation between the destructive and non-destructive test results. Such correlations can be used to calibrate

the NMR system for non-destructive *in situ* measurement of properties of concrete and wood structures in the field.

Principles of Nuclear Magnetic Resonance (NMR)

In this section, a brief summary of the principles of NMR as reported by several researchers (General Electric, 1982; McMurry, 1990; Akkit, 1992) will be given.

The essential elements in NMR measurements are the nuclear spins. Since a nucleus bears an electric charge, its spin produces a magnetic moment, μ . The fields produced by these tiny magnets resemble those of a microscopic bar magnet. In the absence of an external magnetic field the nuclear spins of the magnetic nuclei are oriented randomly (McMurry, 1990). The nuclei, however, orient themselves in specific directions when placed between the poles of a strong magnet with a magnetic field, B_0 . A spinning nucleus can orient itself in two possible directions: (a) parallel to the external magnetic field, where the tiny magnetic field of the nucleus is aligned in the same direction as the external field; and (b) antiparallel, where the nucleus' magnetic field adopts a direction opposite to that of the external field. These possible orientations are illustrated in Figure 1. The orientations shown in Figure 1 do not have the same energy levels and so are not present in equal amounts. The parallel orientation is slightly lower in energy, making this spin-state slightly favoured over the antiparallel orientation. The origin of the NMR technique lies in these energy differences.

An NMR spectrum is obtained by placing a sample in a strong magnetic field and recording the radio frequency (RF) radiation from the nuclei in the sample. If the nuclei are irradiated with electromagnetic radiation of proper frequency, energy is absorbed and a nucleus may get aligned either in the parallel or anti-parallel direction with respect to the applied magnetic field. The nucleus is said to be in resonance with the applied radiation, hence the name nuclear magnetic resonance.

The amount of RF energy required for resonance depends both on the strength of the magnetic field and on the type of the nucleus

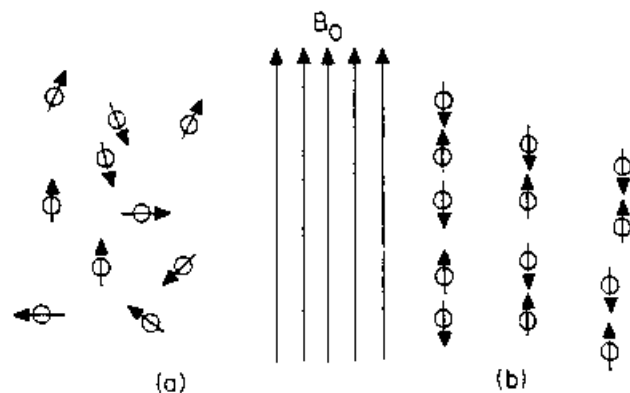


Figure 1. Nuclei orientation: (a) in the absence of external magnetic field; (b) in the presence of external magnetic field

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being irradiated. If a very strong magnetic field is applied, the energy difference between the two spin-states (parallel and antiparallel) is large, and a higher frequency (higher energy) is required for a spin-flip to occur. If a weaker magnetic field is applied, less energy is required to effect the transition between nuclear spin-states.

Without creating resonance, there would be no detectable NMR signal. Resonance is the induction of transitions between two different energy states. The energy required to produce a transition in NMR is the difference in the magnetic energy between the lower and upper base states.

Resonance occurs when radio frequency is applied at the precessional frequency also known as the *Larmor frequency*, flipping the magnetic moments from their parallel (lower energy) to their antiparallel (higher energy) levels (General Electric, 1982). The Larmor frequency (ω_L) is defined by:

$$\omega_L = \gamma B_0 \dots\dots\dots (1)$$

where:

γ = magnetogyric ratio - a fundamental physical constant for each nuclear species; and B_0 = intensity of magnetic field.

As was stated earlier, the spinning of nuclei produces magnetic moments. Quantum mechanics shows that for a proton the spin does not align exactly with the external magnetic field, but rather tilts at an angle, θ , as shown in Figure 2. The magnetic dipoles then begin to precess about the direction of the external field. The precessional frequency depends on the type of nucleus. The precessional movement of the magnetic moment vectors form the surface of a double cone as shown in Figure 3. Their joint alignment creates a macroscopic magnetisation or magnetic moment, M . The nucleus which, because of its large abundance and its favourable magnetic moment, lends itself best to NMR studies is the proton (General Electric, 1982).

Magnetic resonance signals can only be received if transverse magnetisation, that is magnetisation perpendicular to B_0 , is created, since it is this transverse component, M_{xy} , which is time-dependent and thus, according to Faraday's law of induction, can induce a voltage in the receiver coil. Transverse magnetisation is created if a radio frequency field of amplitude B_1 in resonance with the precessing spins, is applied in a direction perpendicular to the main field. If the duration of the B_1 field is such that the entire magnetisation is rotated by an angle of 90 degrees, it will become transverse, that is perpendicular to the static field, B_0 (General Electric, 1982).

Before a system is perturbed by a radio frequency, all the magnetisation occurs along one axis, let us say the Z axis, as shown

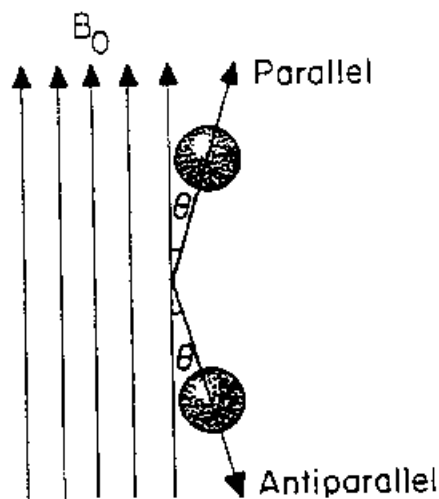


Figure 2. Inclination of magnetic moment with respect to external field

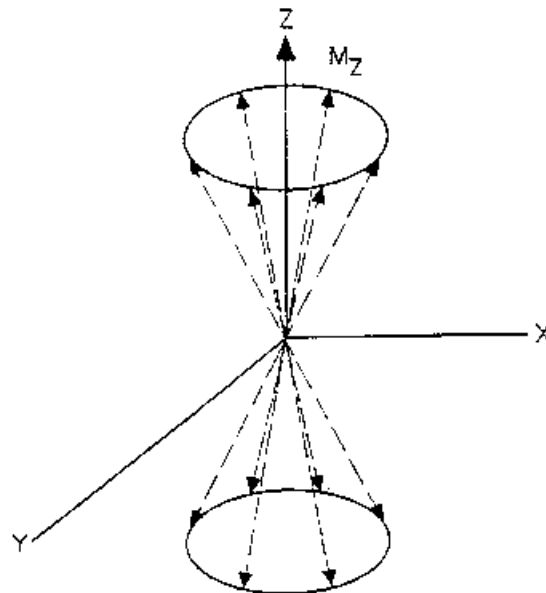


Figure 3. Precessional motion of magnetic moments. Due to random phases, transverse components of individual spins cancel out

in Figure 3. At this stage, the transverse components of the magnetic moment cancel each other, i.e. $M_x = M_y = 0$. But in order to obtain resonance, the system must be perturbed. This is done by passing an RF alternating current through a coil wrapped around the sample space placed along the y-axis, as shown in Figure 4. At this time, the nuclear moments do not have the same components in the XY-plane. Therefore, they do no longer cancel out, and there is a resultant magnetisation M_{xy} transverse to the external field (Akkit, 1992). The magnetisation is no longer static, and the rotating vector, M_{xy} , induces an RF current in the coil around the sample which can be measured as a signature for the material being tested. It is noted that the net magnetisation is of interest, i.e. the magnetisation for the whole sample.

Relaxation process

If a system in equilibrium is perturbed, and if upon removal of the perturbing influence the system is returned to its original condition, the system is said to have *relaxed*. The relaxation normally is not instantaneous, but takes a finite amount of time.

In NMR measurements, the relaxation can be described by a characteristic time, T . If the relaxation is fast, T is small; on the other hand if the relaxation is slow, T is long. The relaxation behaviours of nuclear spins show up directly in their NMR spectra, and are related to the dynamics of the system. Therefore, relaxation times are extremely important in studying the physical and chemical properties of a system.

Spin-Lattice Relaxation Time (T_1): The parameter, T_1 , gives the time of decay of magnetisation in the direction of the applied external magnetic field, B_0 , after excitation. Therefore, T_1 is the relaxation time of magnetisation M_z in the longitudinal direction. Applying a radio frequency pulse, B_1 , can swing the entire nuclear magnetisation away from its equilibrium position in the Z direction (Akkit, 1992). This constitutes the perturbation of a system as discussed above. A 180° pulse will swing the magnetisation vector, M_z , in the opposite direction, as shown in Figure 5. The Figure shows the inversion of magnetisation. Immediately following the end of the pulse, relaxation begins and the magnetisation will go back to its original value and direction. This is known as *longitudinal relaxation* and the characteristic time for this process is T_1 .

Spin-Spin Relaxation Time (T_2): If a 90° pulse is used instead of a 180° pulse to excite the system, the magnetisation vector M_z will

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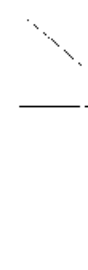


Figure 5.

a 180° RF pulse along X, τ seconds after the initial 90° degree pulse. The nuclear spins continue to precess, but after another τ seconds they will come into phase and the transverse magnetisation M_x will again reach a maximum.

The 180° pulse is a refocusing pulse and the intensity of the spectrometer output rises following this pulse to a maximum τ seconds after, and then it decays again. Subsequent 180° pulses will refocus the magnetisation, which could be refocused indefinitely if the applied pulses were perfect. The refocusing does not work for the random relaxation process. As a result, the duration of the experiment is limited by intrinsic transverse relaxation. In fact, the echoes decay in intensity at a rate determined by the real T_2 , which can be measured from a plot of intensity versus time. The significance of these echoes will become more clear in subsequent sections on applications of NMR where T_2 's are determined for concrete and wood samples.

H-NMR spectroscopy

The hydrogen proton, due to its simplicity and relative abundance, lends itself very well to NMR spectroscopy. In fact, it is often given its own branch of NMR called *proton* or *H-NMR spectroscopy*. The intensity or height of the signal is proportional to γ^2 , where γ is the magnetogyric ratio. The hydrogen proton has the second highest magnetogyric ratio known and therefore this explains why it is so popular for NMR experimentation.

Chemical shift

Another phenomenon that needs to be referred to is called the *chemical shift*. Without this phenomenon, the application of NMR would be limited. Different nuclei would resonate at different frequencies due to variation in γ , but these differences would be hard to quantify. The chemical shift arises because of the way that the electrons shield or screen the nuclei from the external magnetic field. This shielding can be quantified. Furthermore, electron motion can be related to the position of a nucleus in a molecule. It follows that the position of a nucleus in a molecule can then be related to the electron shielding. As a result, the chemical shift causes the NMR signal to behave slightly differently for nuclei in different chemical environments. It is this slight difference, for example, that makes it possible to distinguish protons in free water molecules from protons in bonded hydrogen in wood and concrete. These differences exhibit themselves in NMR signals as will be demonstrated in subsequent sections.

Evaluation of concrete and wood properties using NMR

As was indicated in the preceding section, NMR is particularly suitable for distinguishing the amount of hydrogen in free water and hydrogen chemically-bonded in concrete and wood. This property of NMR will be used and related to mechanical properties of concrete and wood as described in the following sections.

a) Concrete

Early-age strength of concrete is critically dependent upon the rate of hydration of cement or, in other words, the chemical reaction between the water and the cement.

Schreider and MacTavish (1985) studied the spin-lattice relaxation time of hydrated cement pastes. The study focused on monitoring of exchangeable water present in cement pastes. No attention was paid to the water that had entered the structure as a solid hydrate (water that already had chemically reacted with cement). The results obtained allowed a characterisation of the fraction of water protons relaxing toward the lattice with different relaxation times T_1 . It was shown that there are only two magnetisation components present during the hydration process of cement pastes. One was diminishing and the other one was growing.

It was suggested as part of the report that the diminishing magnetisation components represented the liquid like water, and the growing magnetisation components represented the water in the gel phase. No study of concrete, that is a mixture of aggregate and cement, was performed.

Bline *et al* (1988) performed proton spin-lattice magnetisation recovery measurements of exchangeable water in Ordinary Portland Cement (OPC) and white cement. Such measurements allow for an almost continuous monitoring of the evolution of the fractal geometry of cement gels with hydration time. The proton spin-lattice relaxation time (T_1) was measured at room temperature by a standard 90°- τ -90° RF pulse sequence at 50 MHz. Both types of cement pastes were kept in NMR glass tubes. The proton spin lattice relaxation time rate ($1/T_1$) monitors the calcium silicate hydrate (C-S-H) gel production as a function of time, and except in the early stages of hydration, is proportional to the accessible pore surface area of OPC. By monitoring the evolution of $1/T_1$, it was possible to observe the increase in the active gel pore water interface and also the change in the geometry of the newly formed hydration products.

Several other researchers, ie, Lasic and Pintar (1988), Lasic and Corbett (1988), and Rumm and Haranczyk (1991), studied the hydration process of cement using NMR. No study, however, was found to relate the mechanical properties of concrete, such as the compressive strength, to the NMR response.

Sample preparation and testing methods: Two concrete mixes, each with a different water cement (W/C) ratio, were prepared using ordinary Portland Cement Type I-II. The maximum aggregate size was 9.5 mm (3/8 in). The W/C ratio for the two concrete mixes were 0.44 and 0.62. Both mixes were designed according to the PCI Absolute Volume Method (Kosmatka and Panarese, 1990). Two types of tests were performed in this study and the results were correlated: (1) Standard destructive compression tests where 100 mm x 200 mm (4 in x 8 in) concrete cylinders were monotonically loaded to failure; (2) non-destructive testing with NMR. For NMR tests, fresh concrete was placed inside 15 mm x 127 mm (3/8 in x 5 in) test tubes up to a height of 75 mm (3 in).

All samples, ie the cylinders and NMR samples, were made airtight during the curing time by means of paraffin wax to prevent evaporation and to keep the total moisture in the samples constant. In addition, great effort was made to cure all samples at exactly the same temperature and moisture.

The compression strength tests and the NMR tests were performed at the following times (hours) after the cement and the water had been combined to initiate the hydration process: 6, 12, 24, 48, 72, 168, 336, 504 and 672 hours (28 days).

NMR testing: The samples were subjected to a stationary magnetic field (B_0) between the two poles of a magnet and an oscillatory magnetic field (B_1) generated by the RF current circulating through the coils around the sample in a 20 MHz spectrometer, as shown in Figure 7. This RF current also produced a voltage difference between both ends of the coils. This voltage difference, which is the received signal, is referred to as the *intensity of magnetisation* in all plots in subsequent sections.

The total amount of intensity of magnetisation (I_t) present in each sample, was due to the water present in free state (physical water) and the water that had undergone hydration with the cement grains (chemical water). The fraction of the magnetisation that was due to the physical water was labelled I_p , and the fraction of the magnetisation that was due to the chemical water was labelled I_c .

The NMR signal consisted of the free induction decay (FID), which was a fast decay of the intensity of magnetisation. The FID curve, which represented the total magnetisation of the sample, was approximated by a straight line. The result of application of the Carr-Purcell pulse sequences was a series of magnetisation echoes with decreasing amplitude due to the inhomogeneity of concrete sample. The line connecting the peaks of all echo signals, or the echo envelope, was a straight line with a negative slope. The echo envelope

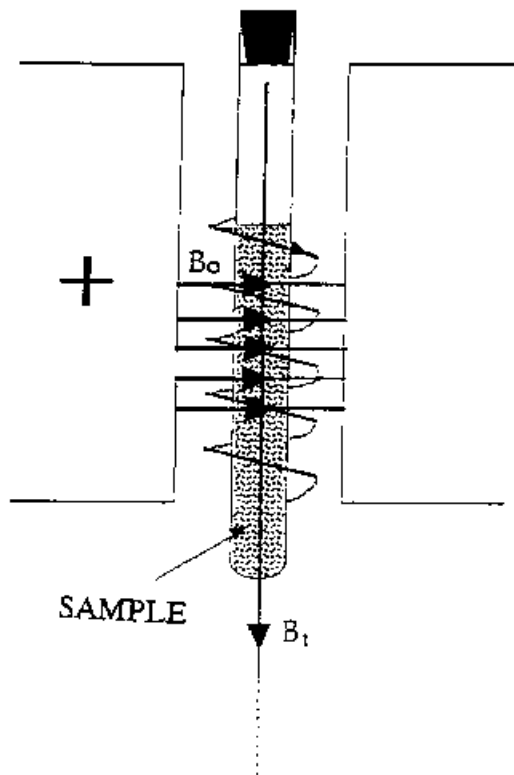


Figure 7. Sample subjected to magnetisation

is the representation of the free water in the sample. Figure 8 shows a schematic of the display generated by NMR testing. In this Figure, the intercept of the FID curve with the vertical axis represents the total magnetisation, I_0 , and the intercept of the echo envelope with the vertical axis represents the magnetisation due to the free water in the sample, I_f . The difference between the two points is the magnetisation due to the hydrated or chemical water, I_c .

Test results: A large number of tests were performed on concrete samples with two different W/C ratios. A brief discussion of sample results are given in the following.

Figures 9a and 9b show plots of percentage of magnetisation of physical, or free, and chemical, or hydrated, water versus time for concrete samples having W/C ratios of 0.44 and 0.62, respectively. As can be seen from these Figures, at early stages of hydration, the

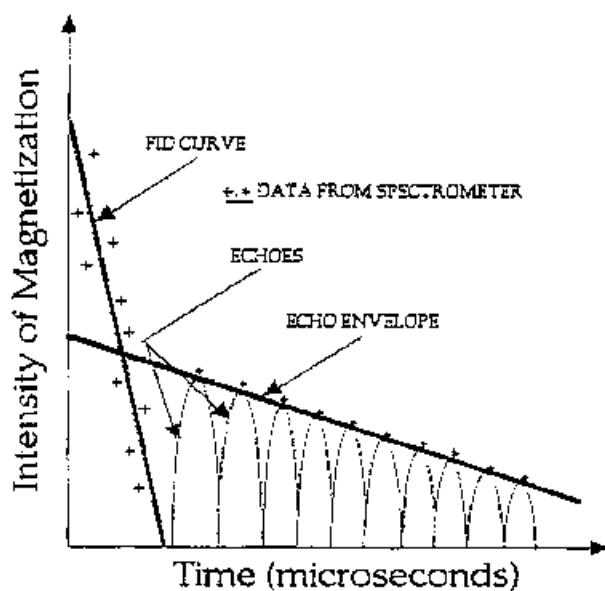
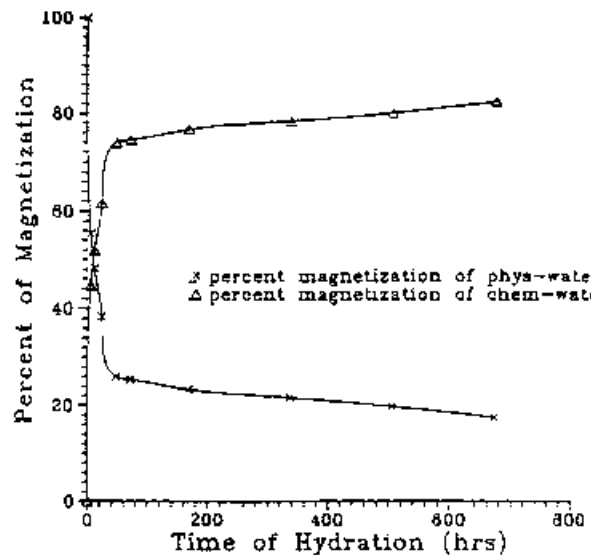


Figure 8. Schematic of display generated by NMR testing

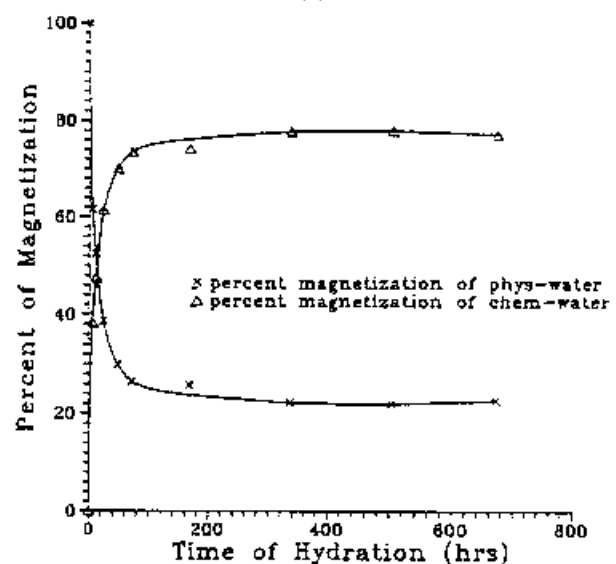
rate of change of physical water to chemical water is significantly higher than at later times, as should be expected. This indicates the suitability of NMR as an effective tool for monitoring the hydration process of cement in concrete. The sum of the percentages of physical and chemical waters at every stage of testing is 100% due to prevention of loss of any moisture by evaporation, etc. All specimens were airtight and completely sealed.

An important mechanical property of concrete is its compressive strength. In order to establish some type of correlation between the compressive strength and NMR response of concrete specimens, a number of destructive compression tests and non-destructive NMR tests were performed at the same time. Three concrete cylinders were tested under compression and the average compressive stress at failure was plotted against percent of magnetisation of the physical water and T_{physical} of the NMR tests.

Figures 10a and 10b show plots of percent of magnetisation of physical water versus compressive strength of concrete for W/C ratios of 0.44 and 0.62 respectively. As can be seen from these Figures, there is a good correlation between the reduction in the amount of physical water and the increase in compressive strength. This correlation is approximated by a straight line, as shown on the Figures. The numbers next to the points on the plots indicate the



(a)



(b)

Figure 9. Variation of percentage of magnetisation with time: (a) W/C = 0.44; and (b) W/C = 0.62

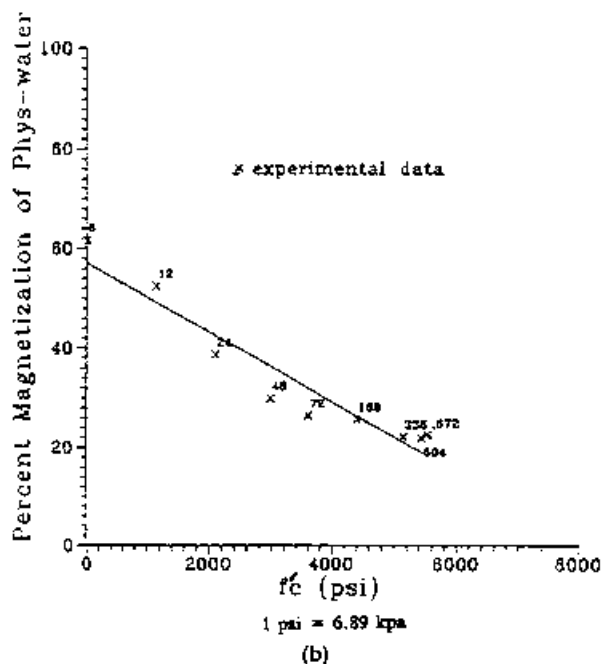
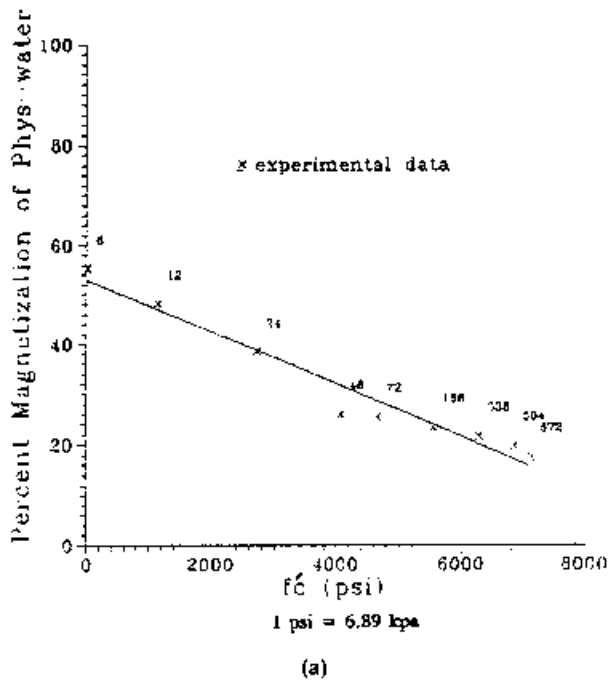


Figure 10. Variation of percent of magnetisation of physical water versus compressive strength of concrete: (a) W/C = 0.44; and (b) W/C = 0.62

time in hours after mixing until tests were performed. There is also a good correlation between the spin-spin relaxation time, $T_{2\text{phys}}$, of the free water in the sample and the compressive strength of concrete, as shown in Figures 11a and 11b for samples with W/C ratios of 0.44 and 0.62 respectively. The curves for both samples were approximated by straight lines. These correlations can be used to calibrate NMR system for *in situ* measurement of compressive strength of concrete in the field.

b) Wood

Because of its suitability for measuring the amount of free or physical water and bond or chemical water, NMR can also be used to monitor the moisture content in wood. It has been well-documented that the strength of wood is related to its moisture content. Therefore, NMR can be used not only to measure the moisture content, but also as demonstrated for concrete, as a non destructive tool for measuring

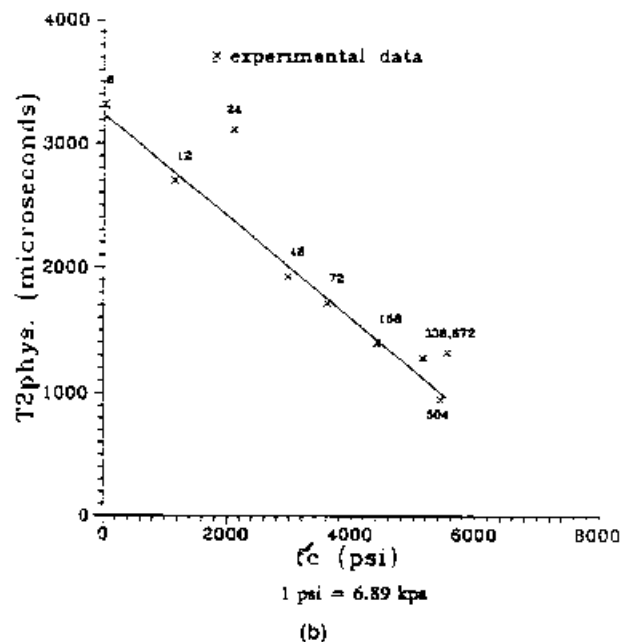
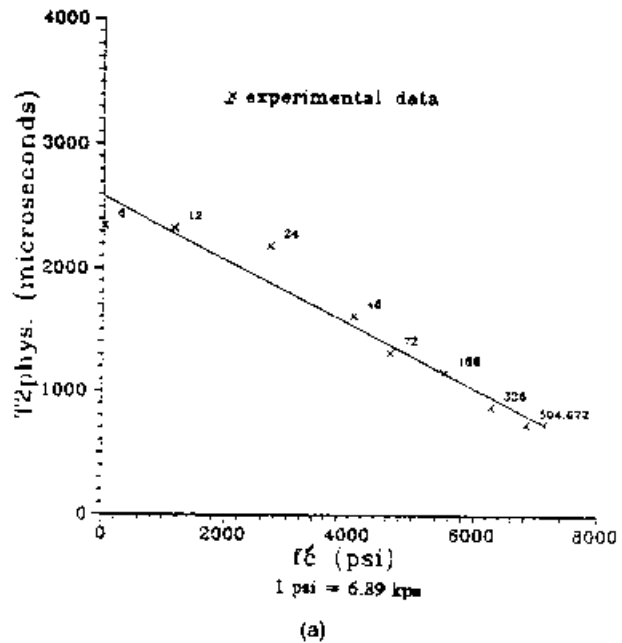


Figure 11. Variation of T_2 of physical water versus compressive strength of concrete: (a) W/C = 0.44; and (b) W/C = 0.62

the strength of the wood. Furthermore, NMR signals will differ for different types of wood, which renders this method effective for distinguishing between different species of wood.

Limited applications of NMR have been reported to examine certain biological properties of wood.

Hibotte *et al* (1990) performed proton magnetic resonance experiments on western red cedar. Specifically, they tested sapwood, heartwood, juvenilewood, and rotten juvenilewood at varying levels of moisture content. They discovered that the signals from the solid wood and the water could be separated and distinguished. Furthermore, they found that the water signal could be subdivided into contributions from different 'water microenvironments' such as earlywood tracheid lumen water, latewood tracheid and ray lumen water, and bound water. These subdivisions were made on the basis of spin-spin relaxation times.

Quick *et al* (1990) studied the drying of western red cedar sapwood with $^2\text{H-NMR}$ imaging by determining the moisture content (MC) and imaging the total water distribution in the wood. The 6 x

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6 x 10 mm (1/4 x 1/4 x 3/8 in) samples were cut with the 10 mm (3/8 in) dimension parallel to the longitudinal tracheids and were imaged using a modified Bruker SXP 4 100 spectrometer operating at 90 MHz. They concluded that H-NMR is an effective new method for observing moisture distribution in wood during drying. They also emphasised the potential of NMR for further research into the physical mechanisms of wood.

Several other researchers (Menon and Macaky, 1987; Araujo *et al.* 1992) have also used NMR to study the water distribution and the cell structure in different species of wood. However, no study was found to relate mechanical properties of wood, such as compression and flexural strengths, to NMR response.

Sample preparation and testing methods: This experiment consisted of two parts:

- (1) destructive testing of wood; and
- (2) non-destructive testing.

Correlations between NMR output, moisture content, and compressive strength were made.

Destructive testing: Compression tests were performed on samples at four 'target' moisture contents (the actual moisture contents at the time of testing was determined by the oven dry method and were slightly different than the targeted values). The target values were 50%, 25%, 15%, and 0%. For each test, at each target moisture content, specimens were tested to examine reproducibility of results.

The compression tests were based upon the recommendations of ASTM 143, 'Standard Methods of Testing Small Clear Specimens of Timber', Part 1. The samples were 50 x 50 x 200 mm (2 x 2 x 8 in) and were oriented with the grain running in the long direction. The specimens were stressed at a constant rate to failure. The load versus deflection over a 150 mm (6 in) gauge length was monitored, as well as the ultimate load at failure. A 25 mm (1 in) long piece of wood near the failure point was removed and the moisture content was determined by the oven-dry method, as specified in ASTM 143.

NMR testing: The non-destructive testing consisted of running two experiments on each specimen using a 20 MHz NMR spectrometer. For each test, two specimens were made and tested to examine the reproducibility of the results. The specimens were approximately 6 x 6 x 90 mm (1/4 x 1/4 x 3 1/2 in), and were placed in NMR test tubes with the grains running in the long direction. These dimensions were required in order for the specimens to fit in the glass test tubes that were used as the sample probe for the spectrometer. At each moisture content, a free induction decay (FID) was obtained from a single pulse experiment, and an echo decay was obtained from a Carr-Purcell sequence. The exact weight of each specimen was measured at each target moisture content in order to determine the actual moisture content. From the FID, the ratio of the physical to chemical water was determined and was plotted versus moisture content.

Test results: Figure 12 shows the plot of the total intensity of magnetisation, I_p , versus moisture content for a sample specimen of Douglas Fir (DF). The total intensity of magnetisation was obtained

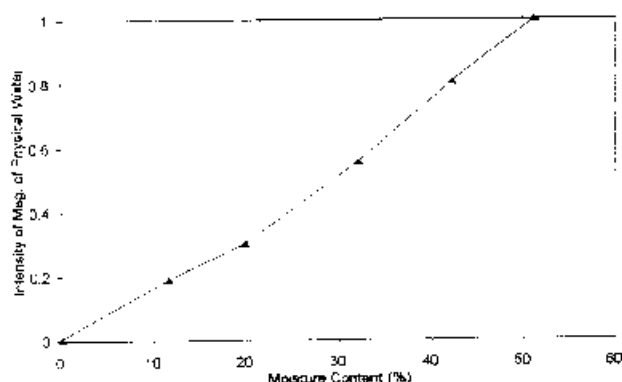


Figure 12. Intensity of magnetisation versus moisture content for a sample of a Douglas Fir

as the intercept of the FID curve with the vertical axis. The intensity of magnetisation was normalised with respect to its maximum value and is shown in percentage form. The actual moisture content of each sample was obtained using the standard oven-dry method. The two results were then plotted against one another (Figure 12). As can be seen from this Figure, there is almost a linear relationship between the total intensity of magnetisation and the moisture content of wood, indicating the potential of NMR for determining *in situ* moisture content of wood structures. Correlation like this can be used to calibrate portable NMR systems for field evaluations of moisture in wood in constructed facilities.

Figure 13 shows the plot of compressive strength of a sample of Douglas Fir versus the intensity of magnetisation of physical water, I_p . As was expected, the compressive strength reduced as the moisture content (free water) in the wood increased. Beyond fibre saturation point, no major change in the compressive strength was observed as the water content increased until the wood sample was fully saturated. Similar observation has also been made when compressive strength of wood is plotted against moisture content obtained using standard oven-dry technique.

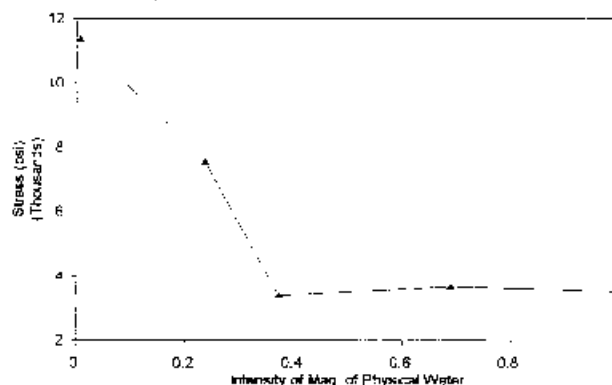


Figure 13. Compressive strength of wood versus intensity of magnetisation of physical water

Conclusions and future studies

The results of tests performed on concrete and wood indicate that there is a potential for non-destructive field evaluation of concrete and wood properties with NMR. This technique can be used to monitor the strength of concrete as it cures. It can also be used to determine the *in situ* moisture content and strength of wood.

The field application of NMR technique, however, requires the development of specialised magnet for inducing the external static magnetic field. It is anticipated that such magnets are feasible and portable spectrometer systems can be developed to accomplish the task of measuring wood and concrete properties in the field. In fact, the NMR system used in the present study was the size of a desk-top computer. Paramagnetic impurities, particularly in concrete, and the effect of steel reinforcement could induce severe interferences in the NMR signals. Such effects need to be filtered out of the results. Currently, studies are being initiated at the University of Arizona to address some of these issues and to pave the way for moving this technology from the laboratory into field applications. The results will potentially have an impact in the area of non-destructive testing of construction materials. The technology can be employed in the overall scheme of infrastructure rehabilitation for more accurate and quantitative evaluation of *in situ* property of construction materials.

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