Non-destructive testing of wood using nuclear magnetic resonance (NMR) spectroscopy

M H Larsen, H Saadatmanesh and M R Ehsani

This paper presents the results of a study utilising NMR to measure moisture content and strength of wood. Different species of wood at different moisture contents were evaluated. Standard destructive tests as well as non-destructive tests using NMR were conducted. Correlations were made between the results of destructive and non-destructive tests. As reported by other researchers also, an almost linear relationship was observed between the moisture content of wood and the intensity of magnetisation. Consistent trends were observed between the stress at failure of wood and the intensity of magnetisation of physical protons.

Introduction

The infrastructure system is an immense network of roads, buildings and bridges that are required to meet basic social and economic needs of the public. The inability of these resources to meet the demands placed upon them poses tremendous problems for the people who use them every day. However, in many instances, the infrastructure is deteriorating to the point where failure is imminent, solutions are lacking and price tags are high.

In order to combat the problem of a dilapidating infrastructure, structures must either be rebuilt or repaired. Before solutions can be offered and implemented, however, the condition of a structure must be determined. Therefore, efficient methods of evaluating the structural integrity of existing buildings and bridges need to be developed. Destructive methods, such as physically removing a sample of a structural component and testing it, are not feasible because they damage the component that is being evaluated. Consequently, methods to examine infrastructure components non-destructively in simple, reliable and efficient ways need to be developed.

Wooden structures, due to their relatively low cost and ease of construction, make up a very large portion of the existing infrastructure. The housing industry alone contributes approximately two million new wooden homes each year in the United States (Goetz, 1989). As a structural material, wood is vulnerable to damage by decay-causing fungi, insects, weathering and chemicals. However, it is often difficult or impossible to determine the inner soundness or weakness of a wood member without destructively testing it.

A potential method to non-destructively examine the physical properties of wood is Nuclear Magnetic Resonance (NMR) spectroscopy. H-NMR is the branch of spectroscopy that deals with the response of hydrogen nuclei to magnetic excitation. From the spectral signals recorded in an experiment, the relative abundance and even the motional state or 'environment' of the nuclei can be deduced. This information can be directly linked to the moisture content of a wood sample, which for certain conditions can give an indication of the strength and soundness of that sample.

This paper discusses the results of tests conducted on Douglas fir, redwood and mahogany wood samples. Destructive tests - strength in compression - were conducted to determine physical properties of the wood. Non-destructive tests using H-NMR spectroscopy were conducted and correlations were sought for between the NMR parameters and the strength of different species of the wood samples. The comprehensive results of this study are given in a thesis completed at the University of Arizona (Larsen, 1994).

Nuclear Magnetic Resonance spectroscopy

Spectroscopy can be defined as the interaction between matter and electromagnetic radiation such that energy is absorbed or emitted in direct proportion to the frequency of the radiation (Harris, 1986). For years, spectroscopy has been one of the most important and widely used methods available for studying molecular structure and processes. In the branch of spectroscopy called NMR, nuclei are placed in a magnetic field and the resulting magnetic energies of the nuclei are studied and interpreted. As with all types of spectroscopy, an NMR experiment yields spectra that can be described in terms of the frequency, magnitude and shape of lines or bands. These parameters, which describe the energy transitions undergone by the nuclei, can then be linked to the molecular structure and motional state of the nuclei.

The NMR spectrometer probes a sample to the level of the atomic nucleus. Many types of nuclei possess what is termed *angular momentum*. In more simple terms, many nuclei spin like tiny tops. Since nuclei are charged particles, their spinning creates a magnetic field around the nucleus. According to quantum mechanical theory, the total angular momentum of any particle will have only certain discrete values, that is the momentum cannot be any arbitrary value. If the total angular momentum, or spin, changes, it will do so by discrete amounts. The hydrogen proton, due to its relative abundance and favourable properties, is one of the most widely-studied nuclei in NMR spectroscopy. The angular momentum of the proton can be one of only two discrete values. In other words, it exists in only one of two 'spin states'.

In general, the directions of the spins of nuclei will be oriented randomly. However, when placed in an external magnetic field, the nuclei will all try to line up with the direction of that external field. In the case of protons, the nuclei will be oriented either parallel or anti-parallel (opposite) to the magnetic field, corresponding to the low- and high energy states, respectively. Although the spinning protons attempt to line up with the magnetic field, they never actually achieve this. The protons are pulled toward the direction of the external magnetic field, but precess about it instead. This motion can be thought of in terms of a spinning top. The top will never line up perfectly with the force of gravity, but will wobble slightly about it (precession). The top would wobble faster if the gravitational force were increased, just as the proton will precess faster if the magnetic

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field is increased. The rate of precession depends also on the type of nucleus being studied.

It is at this point that the phenomenon of resonance can be understood. If an additional weak magnetic field, B_1, is applied in a direction perpendicular to the external magnetic field, B_0, then it will also apply a force on the magnetic fields of the nuclei. Applying the B_1 field such that it rotates at exactly the same speed as the precession of the nuclei being studied will cause the nuclei to flip to another spin state. This is the resonant condition and it corresponds to a release of energy that can be monitored and analysed. Without creating resonance, there would be no detectable signal.

The power of NMR lies in the information that can be deciphered from the resulting signal. After a sample has been subjected to an NMR pulse, the signal given off by the nuclei being studied will decay to zero. The signal output plotted *versus* time is termed a *free induction decay*, or FID (see Figure 1). The rate of decay can be described by two time constants - the spin-lattice relaxation time, T_1, and the spin-spin relaxation time, T_2. The characteristics of the FID and the values of the two time constants are used to measure the concentrations of structurally different types of nuclei present.

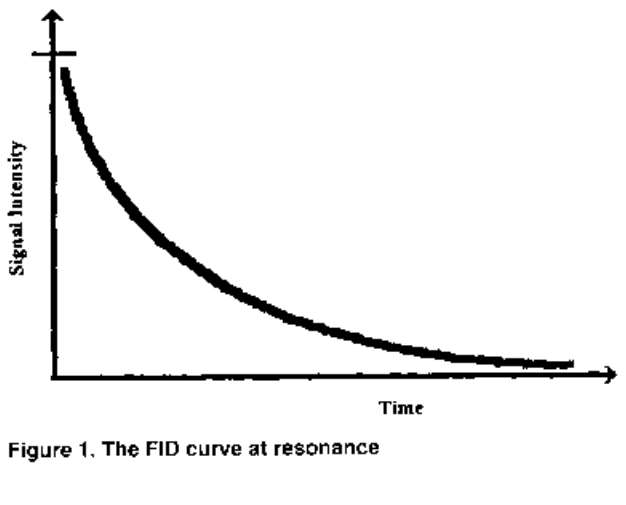


Figure 1. The FID curve at resonance

Previous studies

Several researchers have investigated the physical and chemical properties of wood using NMR spectroscopy.

Menon *et al* (1987) used proton magnetic resonance to study water in Douglas fir and western red cedar. The free-induction decay (FID), T_1 and T_2 were measured at varying moisture contents as the wood samples were continually analysed, dried and re-analysed. It was shown that the NMR signal of water was easily distinguishable from that of the wood. Using the fact that the NMR signal is proportional to proton density, the moisture content of the wood was obtained from the FID without using a reference standard. Proportionality constants for the relationships between NMR signal and moisture content were determined from the chemical composition of the wood species. The spin-lattice relaxation time, T_1, was found to be noticeably different in the two species. In sapwood samples, three distinct spin-spin relaxation times, T_2, were recognised and found to correlate with water in and on the cell wall, water in the ray and latewood tracheid lumens, and water in the earlywood tracheid lumens. In other words, different water 'environments' were distinguishable on the basis of T_2.

Araujo *et al* (1992) utilised two new NMR techniques, relaxation spectra and relaxation selective imaging, to investigate the water distribution in white spruce. Samples of normal white spruce sapwood, heartwood and juvenilewood as well as two rehydrated heartwood samples, one containing incipient decay and one compression wood, were analysed using NMR spectroscopy. The moisture content of the samples was calculated from NMR data using the same method employed by Menon *et al*. The authors found that since the NMR signal is proportional to the number of protons, the distribution of T_2 relaxation times for lumen water should reflect the distribution of cell radii by volume. Consequently, it is more appropriate to analyse the T- decay curve in terms of a continuous distribution of T_2 values rather than the conventional interpretation in terms of a fixed number of discrete components. This relaxation spectra technique seemed to work extremely well. It was found that the continuous T_2 spectrum consisted of a series of large peaks. The area under each peak corresponds to the amount of moisture in a particular water environment; the T_2 value indicates the nature of the environment; and the shape of the spectrum reflects the radial distribution of the cells in the sample. It was further found that normal sapwood could be distinguished from normal heartwood and juvenilewood since the latter two contains almost no lumen water. Incipient rot could not be distinguished from normal wood. However, compression wood was readily distinguishable from normal wood when the lumens are hydrated because of the very different size distribution of the cell radii.

Pearson and Rhyti (1990) introduced a method that can be used in production to measure the moisture content of wheat. They used a spectrometer which had been modified specifically to withstand the harsh conditions of the production line. The spectrometer is small, powerful and is resistant to the dusty, humid environment of a flourmill. The authors tested dry wheat and freshly tempered wheat. They distinguished two portions of the NMR signal decay: a rapidly decaying portion, which they termed the *chemical hydrogen signal*, and a slowly decaying portion, which they termed the *physical hydrogen signal*. The chemical portion corresponds to the free water in the wheat. The ratio of the chemical to physical signal was used as a measure of the moisture content of the wheat sample. Plotting the physical/chemical ratio *versus* the actual moisture content of the wheat yielded very good straight-line results. Once calibration curves were developed for various moisture contents, the moisture content of any wheat sample could be determined using the NMR spectrometer.

Experimental study

The experimental procedures for this project were as follows. Destructive tests (compression) were performed in order to verify the relationships between moisture content and strength, and between species and strength. Next, an NMR analysis was conducted on the same wood material in order to search for relationships between NMR output and moisture content. Figure 2 shows the NMR test specimen in a test tube placed between the magnets in a portable NMR device. Finally, correlations were sought for between the destructive and the non destructive tests in order to find reasonable ways to relate the NMR output to the strength and species of the wood.

Three species of wood were used: Douglas fir, redwood, and mahogany. Boards were handpicked from a local supplier in order

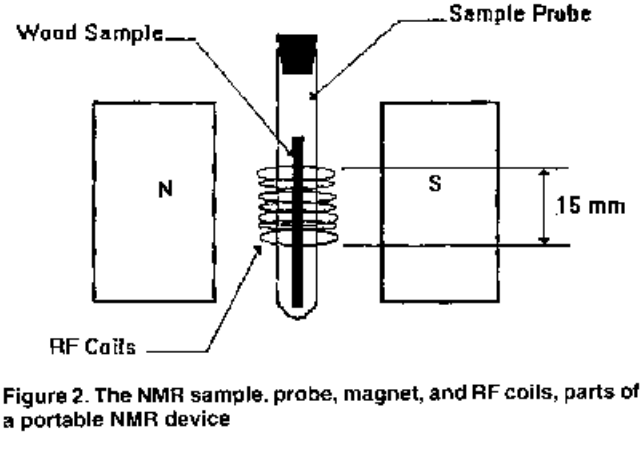


Figure 2. The NMR sample, probe, magnet, and RF coils, parts of a portable NMR device

to find straight grained wood with a minimal amount of knots and other defects. Boards of Douglas fir and redwood were 4 in x 4 in x 16 ft (10.2 x 10.2 x 487.7 cm) nominal size. The board of mahogany was 2 in x 12 in x 6 ft (5.1 x 30.5 x 182.9 cm) nominal size. These were the largest size boards that could be obtained locally. All test specimens (bending, compression, and NMR specimens) for a species were taken from a single board to limit as much as possible any variations in the wood stock.

Destructive tests

For each species, compression tests were performed on separate samples at four 'target' moisture contents (the actual moisture contents at the time of testing were determined by the oven dry method and were slightly different than the targeted values). The target values were 50%, 30%, 10% and 0%. For each test at each target moisture content, two specimens were tested to examine reproducibility of results. Therefore, the destructive evaluation of each species consisted of eight compression tests.

Specimen sizes and preparation

The compression tests were based upon the recommendations of ASTM 143, 'Standard Methods of Testing Small Clear Specimens of Timber', Part I (ASTM, 1991). From the boards of each species, eight compression specimens were cut with dimensions 2.25 in x 2.25 in x 8.5 in (5.7 x 5.7 x 21.6 cm) with the grain running in the long direction. All the compression specimens were then placed in an oven at a temperature of 180° F (82° C) until the specimens attained constant weights. The samples were allowed to equilibrate to room temperature under airtight conditions, and were then cut to the appropriate size for compression testing - 2 in x 2 in x 8 in (5.1 x 5.1 x 20.3 cm). This was done in order to bring all the compression specimens to the same moisture content before they were cut to the proper size for testing. This ensured that each specimen, regardless of moisture content, contained approximately the same amount of wood material. If the specimens had been brought to the target moisture content and then sized for testing, each specimen would have a different amount of wood material, due to the effects that water has on the dimensions of the wood.

The exact dimensions and weights of each sample were recorded. The samples were then hydrated by soaking them in water in airtight bags. By weighing the samples, an estimate of the moisture content at any time could be made in comparison to the initial weight of the specimen. Each specimen was hydrated to an estimated moisture content slightly above the target moisture content and was then allowed to equilibrate in an airtight container. The compression specimens were allowed to equilibrate for approximately eight weeks. This equilibration period was utilised to allow the moisture to be uniformly distributed throughout the samples. Before testing, the ends of the compression specimens were trimmed slightly to eliminate checks and to square up the surfaces.

Testing equipment and methods

The destructive tests were performed on a Tinius Olsen machine. This testing machine was equipped with a floating platen head to compensate for uneven surfaces of test specimens. The compression tests were performed according to ASTM 143. The specimens were loaded at a constant rate to failure and the deformation over a six-inch gauge length was measured via a dial gauge attached to the specimen. Throughout the tests, the load *versus* deformation was recorded.

After each test, the actual moisture content of the specimen was determined as specified in ASTM 143. A 1-inch (2.54 cm) section of the specimen was extracted near the location of the failure. The section was weighed (Wet Weight), was placed in an oven at approximately 200° F (93° C), and was dried to constant weight (Dry Weight). The moisture content (MC) was determined as:

$$MC = \frac{\text{Wet Weight} - \text{Dry Weight}}{\text{Dry Weight}} \quad \ldots\ldots\ldots\ldots (1)$$

Non-destructive tests using NMR

The non-destructive testing consisted of running two experiments on each specimen using a 20 Hz NMR spectrometer. For each species, two specimens were made and were tested to examine the reproducibility of the results. The specimens were tested at seven different target moisture contents: 60%, 50%, 40%, 30%, 20%, 10% and 0%. At each moisture content, a free induction decay (FID) was obtained from a single pulse experiment, and an echo decay was obtained from a CPMG sequence.

Specimen sizes and preparation

As stated previously, specimens for NMR analysis were taken from the same wood stock as the destructive tests. Six specimens were made - two Douglas firs, two redwoods, and two mahogany samples. The specimens were 0.25 in x 0.25 in x 3.5 in (.65 x .65 x 8.9 cm) and were oriented with the grain 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. The samples were treated in exactly the same manner as the destructive specimens. They were cut slightly oversize initially, and were dried to constant weight at approximately 180°F (82 C). They were then cut to final dimensions and were weighed. The specimens were hydrated to approximately 60% moisture content by soaking in water. The moisture content at any point during the experiment was estimated by weighing the sample and comparing the weight to the initial weight. The specimens were allowed to equilibrate overnight and were kept in airtight, capped test-tubes.

After testing in the spectrometer, each specimen was weighed and was allowed to air-dry for several minutes until the estimated moisture content had been reduced to the next lower target moisture content. The specimens were again allowed to equilibrate overnight before the next round of tests was performed. This process was repeated until the moisture content had been reduced to 0%, which required the specimens to be oven dried. The actual moisture content of the specimens at each stage of the experiment was then calculated using the weight at the time of testing and the oven-dry weight.

Testing equipment and methods

The non-destructive tests were performed on a custom-built NMR spectrometer running at 20 MHz. The spectrometer was connected to a NEC personal computer. All operations were initiated from the keyboard and data digitisation and storage was accomplished by the PC.

Once the resonant frequency was established for the hydrogen proton, a wood sample was placed in the probe and inserted into the NMR spectrometer. Two experiments were performed on each sample. A single-pulse experiment was performed in order to measure the free induction decay (FID) of the sample. Next, a multiple-pulse, or echo, experiment was performed in order to measure the spin-spin relaxation time (T_2) of the sample. The echo sequence is called the Carr-Purcell-Meiboom-Gill (CPMG) sequence.

Analytical methods

Analysis of destructive tests

For the compression tests of the wood samples, the deflections corresponding to incremental loading were monitored throughout the duration of the test. Load *versus* deflection curves were then plotted from the data collected.

The goal of this part of the experiment was to verify the relationship between the moisture content of a specimen and the strength of that specimen. Consequently, the ultimate load at failure

for each specimen was noted. For each species of wood, the ultimate load at failure was plotted *versus* the moisture content of the specimen. Results of tests performed by other researchers. Forest Service Bulletin 70 (Tiemann, 1906), for example, have established linear relationships between ultimate load and moisture content for wood tested in bending and compression. At high moisture contents, the ultimate load is constant. As wood is continually dried, there is no change in ultimate load until a moisture content around 20-30%. This is typically called the 'fibre saturation point' and is thought to correspond to the point when all the free water is extracted from the wood sample and only water in the wood cells remain. Further drying below the fibre saturation point results in an increase in ultimate load. The ultimate load increases linearly until it reaches a maximum at 0% moisture content. Figure 3 depicts a moisture content *versus* load curve for compression tests on Douglas fir. The two linear portions of the curve are readily apparent.

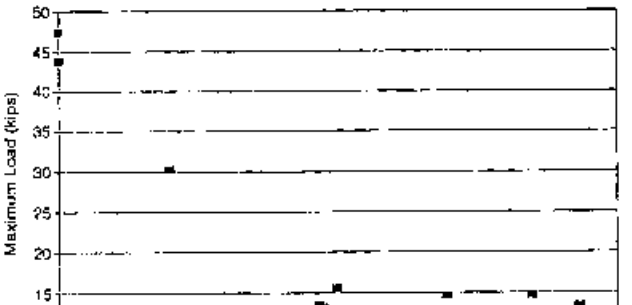


Figure 3. Moisture content *vs* ultimate load for a sample of Douglas fir

Analysis of NMR tests

As described earlier, two NMR experiments were conducted on each wood specimen - a single-pulse FID experiment and a CPMG echo sequence. The output consisted of files of digitised data points, which represented the signal intensity throughout the time duration of the experiment. These data points yielded the FID curve and the echo curves.

Figure 4 shows a typical FID curve for a wood sample. Two portions of the graph are readily apparent - a quickly decaying portion due to the wood protons, and a slowly decaying portion due to the free water in the sample. This is exactly what was expected considering the previous experiments described in the literature review.

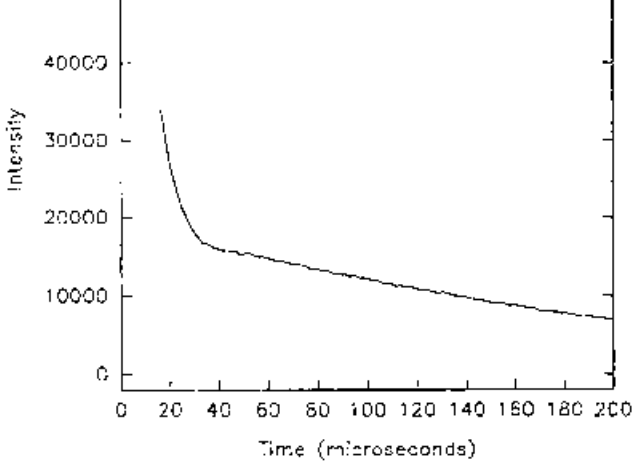


Figure 4. Typical FID curve for a wood sample

From the equation that describes the signal intensity of magnetically excited protons, it can be shown that the initial signal intensity is directly proportional to the number of protons that were excited. Therefore, the initial signal intensities of the chemical (wood), and the physical (free) protons would yield the relative proportions of each type of proton. However, the initial signal intensities cannot be determined from the graphs. During the first 10 to 15 µs after the radio frequency (RF) pulse is turned off, the receiver electronics are saturated due to the enormous signal generated by the pulse. Consequently, no meaningful data can be recorded during this time. This block of time is usually termed the 'dead time'.

An estimate of the initial signal intensities can be made by extrapolating the respective curves to their zero-time intercepts. However, the question of what type of extrapolation should be used is immediately encountered. From quantum mechanical theory, it can be shown that the decay of the NMR signal is exponential. However, in a free-induction-decay, the effects of magnetic inhomogeneities cause the signal to decay much more quickly than is expected. Previous researchers have fit straight lines through the quickly decaying portion of the curve and through the first part of the slowly decaying portion of the curve (which is very close to being linear). This yields a fairly reasonable estimate of the initial intensities. For this experiment, a linear fit was made to the quickly decaying portion of the graph from time t = 16 µs to t = 21 µs. Another linear fit was made to the relatively straight part of the slowly decaying portion of the graph from time t = 60 µs to t = 120 µs. The data was fitted to the equation:

$$y = mx + b \quad \ldots\ldots (2)$$

using linear regression incorporated in SigmaPlot 4.1 software (Jandell, 1991). The zero-time intercepts are the y-intercepts of the linear regression.

The echo curve

The multiple-echo pulse sequence is utilised in order to eliminate the effects of magnetic inhomogeneities so that the true decay of the magnetic energy can be measured. It should be remembered that after the refocusing pulse is applied and the phase coherence is regained, the output signal will grow and will peak at regular intervals. The peaks of these echoes represent the true level of magnetisation at that time. Therefore, connecting the peaks of many successive echoes will produce the true decay of the magnetic signal. The results of an echo pulse sequence are shown in Figure 5. The effects of the refocusing pulses can be seen between the echo peaks. Figure 6 shows echoes at an enlarged scale.

In order to create the echo envelope, the data files were reviewed and the echo peaks were observed from inspection. The signal

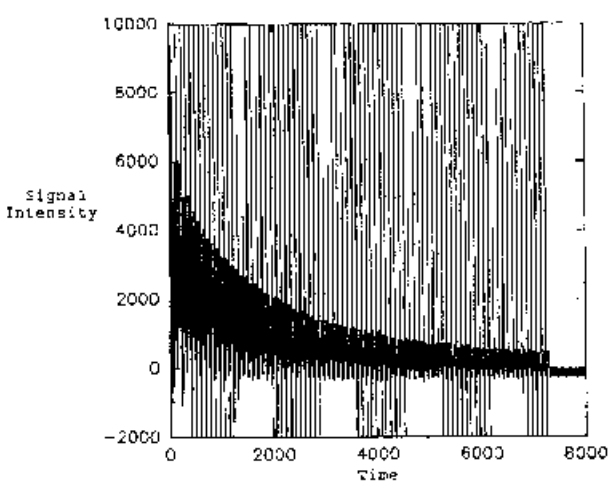
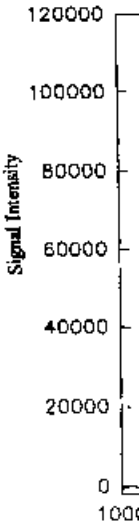


Figure 5. The echoes formed by a CPMG sequence



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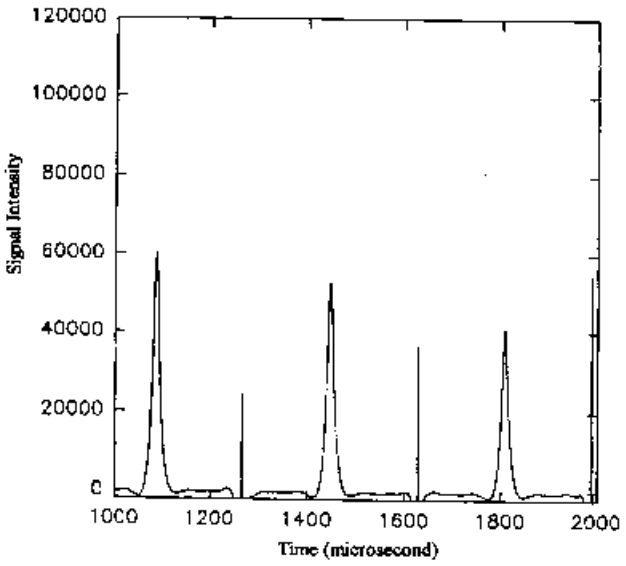

Figure 6. Echoes at an enlarged scale

intensity and the time at the echo peaks were recorded and were plotted. This decay is, of course, exponential. In the literature review of other researchers, it was explained that three exponential curves can be extracted which correspond to hydrogen protons in the three water 'microenvironments'.

The signal from the wood protons dies out very quickly due to the highly structured state of these protons. In fact, the signal from the wood protons decays in approximately 30 to 50 µs. In looking at the CPMG echo pulse sequence it is noted that the delay time between the 90° pulse and the first 180° pulse is 400 µs. Therefore, the signal from the wood protons has totally decayed before the first echo peak is formed. As a result, the echo decay curve provides information about the relaxation of the water protons that are contained in the wood sample, but cannot provide information about the relaxation of the wood protons.

A tri-exponential curve was fit to each echo envelope. The Marquardt-Levenberg algorithm was utilised to fit the curves to the equation:

$$y = a \cdot \exp(-b \cdot x) + c \cdot \exp(-d \cdot x) + e \cdot \exp(-f \cdot x) \quad \ldots\ldots (3)$$

As discussed by Menon *et al* (1987), the components of the curve can be assigned to water in and on the cell wall, water in the ray and latewood tracheid lumens, and water in the earlywood tracheid lumens. Each component has a separate, distinct T_2 value which, upon inspection of the tri exponential, can be calculated as 1/a, 1/b and 1/c, respectively. The three T_2 values are usually abbreviated as the fast, medium, and slow T_2s. As a wood sample is dried, water will be lost from the components, and the a, b and c values will reduce. Values of T_2 should theoretically remain relatively constant. However, this has not always been shown to be the case at moisture contents below about 80% (Menon *et al*, 1987).

Test results

Correlations have been sought for between the two types of tests in order to gauge the applicability of NMR testing methods to the determination of the mechanical properties of wood samples.

Destructive tests

For each species, the ultimate load at failure was converted to ultimate stress at failure. For the compression tests, stress was calculated as load divided by cross sectional area of the sample. The ultimate stress at failure was plotted *versus* the moisture content of the sample for each test. Typical results are shown in Figure 7.

In the compression tests, the strong dependence of the ultimate

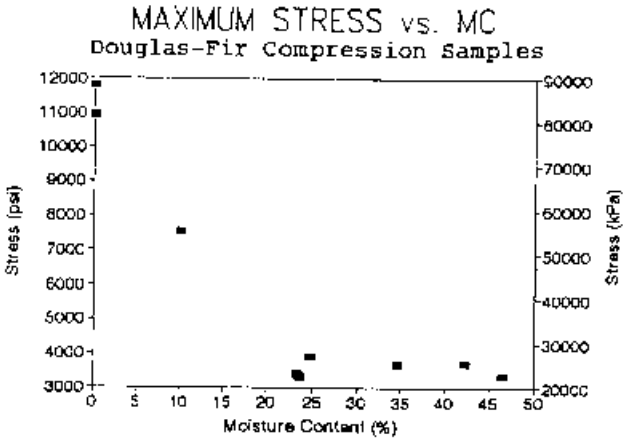

Figure 7. Typical relationship between ultimate compressive stress and moisture content for Douglas fir

load upon the moisture content is readily apparent. At high moisture contents, the strength of the specimens for each species is constant. At approximately 25-30% moisture content, however, this changes. Further drying results in higher ultimate loads. The relationship between moisture content and strength in this region appears to be linear. This is very much in agreement with the results of previous researchers (Tiemann, 1906).

Non-destructive tests

As discussed earlier, the NMR testing yielded two types of output - FID curves and echo curves. For each species of wood, two identical specimens were tested at the same moisture content in order to get an indication of the reliability and reproducibility of the results.

For each of the FIDs, a bi-linear fit to the data was performed and the following parameters were calculated:

- T(0) . . . . total initial signal intensity of the FID (applicable only to the bi linear fit).
- P(0) . . . . initial signal intensity of physical protons.
- C(0) . . . . initial signal intensity of chemical protons.
- R . . . . ratio of physical to chemical protons, R=P/C.

From the echo curves, a tri-exponential fit was performed on the echo envelope (the peaks of the echoes). The following parameters were calculated:

- T_2(fast) . . . . spin-spin relaxation time of the tightly bound water protons.
- T_2(med) . . . . spin-spin relaxation time of the loosely bound water protons.
- T_2(slow) . . . . spin-spin relaxation time of the free water protons.

Each of the calculated parameters was analysed to determine whether relationships existed between the parameter and the physical properties of the wood (such as mechanical properties or moisture content). From the FID curves, it was observed that the following parameters exhibited relationships with the moisture content of the wood: the intensity of magnetisation of the physical protons P(0), the intensity of magnetisation of the chemical protons C(0), and the ratio of the physical to chemical protons R. From the echo curves, it was observed that the T_2(fast) parameter was related to the moisture content. The T_2(med) and T_2(slow) parameters were not present at lower moisture contents.

In order to compare the results of the FIDs, several graphs were developed. For each wood sample, the following graphs were plotted: the intensity of magnetisation of physical protons was plotted *versus* moisture content, the intensity of magnetisation of chemical protons was plotted *versus* moisture content, and the ratio of physical to chemical protons was plotted *versus* moisture content. The values

of P(0), C(0), and R have all been normalised with respect to the initial value at the highest target moisture content. Linear regression was performed on each set of data, and a 'best fit' line was plotted for each graph. It should be remembered that the signal intensity of the physical protons refers to the amount of physical (free) water in the sample. The signal intensity of the chemical protons refers to the amount of chemically bound hydrogen in the wood material itself. The ratio of these parameters should yield an estimate of the relative abundance of each type of hydrogen, and hence should yield an estimate of the moisture content of the sample. Figures 8 through 10 show typical plots of moisture content *versus* intensity of magnetisation of physical protons, intensity of magnetisation of

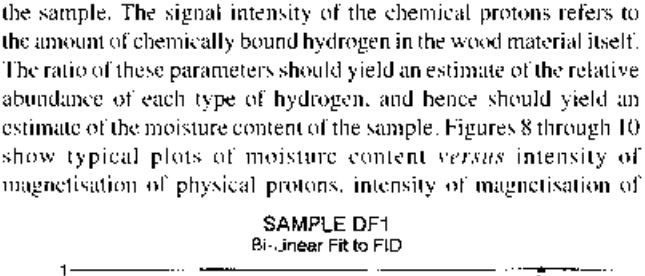

Figure 8. Plot of intensity of magnetisation of physical protons *vs* moisture content (Douglas fir)

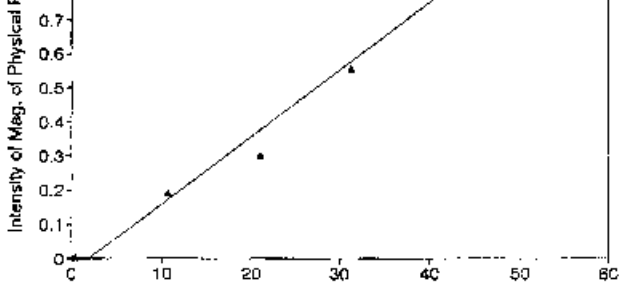

Figure 9. Plot of intensity of magnetisation of chemical protons *vs* moisture content (Douglas fir)

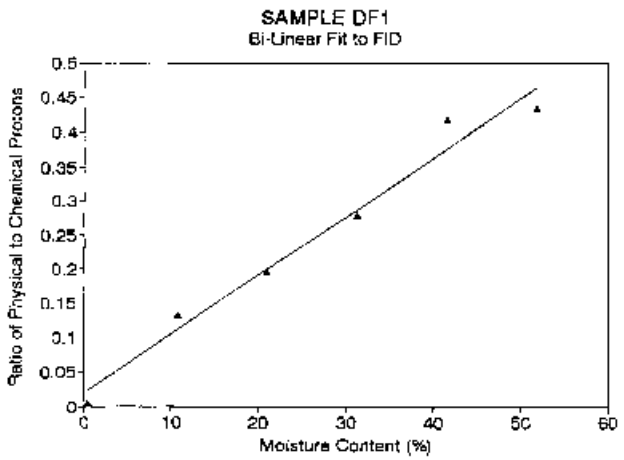

Figure 10. Plot of ratio of physical to chemical proton intensity *vs* moisture content (Douglas fir)

chemical protons and the ratio of physical to chemical protons, respectively.

In comparing the graphs, it is obvious that the intensity of magnetisation of physical protons has a linear relationship with the moisture content. The R-squared values for these graphs are consistently in the 0.98-0.99 range for the bi-linear fit to the FIDs. The effects of drying a wood sample can therefore be accurately monitored by NMR analysis. As water leaves the sample, the intensity of magnetisation of the physical protons diminishes proportionally. In other words, an NMR spectrometer can monitor the loss of free water as the physical protons decrease in abundance.

The intensity of magnetisation of the chemical water is somewhat more unpredictable. The bi-linear fit to the FIDs yielded R-squared values above 0.9. Although this is quite good, it is not nearly as reliable as the relationship between physical water intensity and moisture content. In analysing the graphs, it appears that chemically-bound protons are leaving the sample as the moisture content drops, due to the fact that the intensity of magnetisation of the chemical protons diminishes with decreasing moisture content. This would appear to indicate that some of the actual wood material is being lost in the drying process. This, of course, does not seem possible. A better explanation for this phenomenon is that part of the signal from the chemical protons is actually due to 'free' water, which is very tightly bound in the cell structure of the wood molecules. Since the water is so tightly bound, it exhibits much faster relaxation times and therefore behaves very much like the chemically-bound protons in the wood molecules.

The relationship between the ratio of physical to chemical protons and the moisture content is, of course, a mix of the preceding results. R-squared values for the bi-linear fit are generally above 0.95. Since the chemical water intensity is included in the ratio, the lower correlation of this parameter obviously affects the correlation of the ratio to the moisture content.

The intensity of magnetisation of the physical protons seems to be the best indicator of the moisture content of the sample. This is a logical result, since the relaxation times for the physical water are longer than for the chemical protons. The chemical proton signal dies out very quickly because these protons are bound rather tightly to the lattice. The physical protons, on the other hand, are loosely bound and have long relaxation times. Consequently, there are many more data points to define the decay of the physical proton signal, which tends to make it a more reliable parameter.

In order to compare the results of the echo tests, the calculated values of T_2(fast) were plotted *versus* moisture content for each of the three species. As noted by other researchers, the T_2 values from the echoes should remain relatively constant, especially at high moisture contents (Menon, 1987). However, Menon *et al.* noticed that at lower moisture contents (below about 80% i, the T_2 values changed and were not constant with moisture content. In this

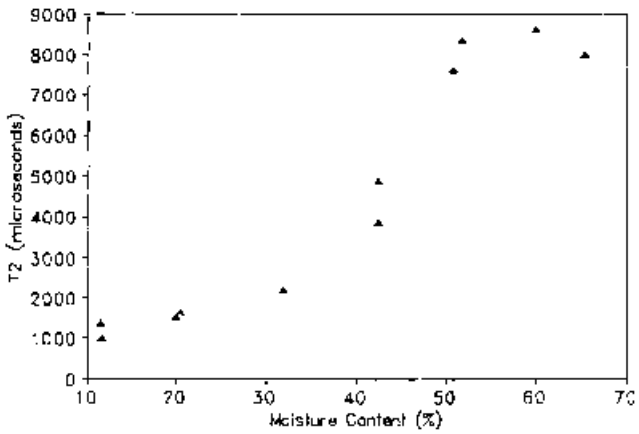

Figure 11. T_2 (fast) *vs* moisture content for a sample of Douglas fir

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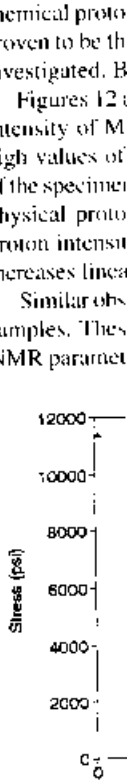

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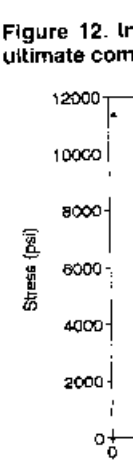

**Figure 13. F
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experiment, the T_2(fast) values decreased with decreasing moisture content, as shown in Figure 11 for Douglas fir. Generally, similar observations were also made for mahogany and redwood samples. For the mahogany samples, the relationship between T_2 and moisture appeared to be linear. More studies need to be conducted in order to determine if this linear relationship is valid, and if it occurs for the other species.

Correlation of destructive and non-destructive tests

In the tests described, it has been shown that the compressive strength of wood can be directly linked to its moisture content. Furthermore, it has been shown that NMR parameters such as the intensity of magnetisation of the physical protons can be directly linked to the moisture content of the sample. It follows, then, that the NMR parameters could be correlated to the mechanical properties of the wood.

In order to show this correlation, several plots have been made. The compressive strength of the wood samples, expressed as ultimate stress at failure, has been plotted *versus* the intensity of magnetisation of the physical protons, and *versus* the ratio of the physical to chemical protons. These parameters were chosen because they were proven to be the most reliable and informative parameters that were investigated. Best-fit lines were plotted through the data.

Figures 12 and 13 show the graphs of Compressive Stress *versus* Intensity of Magnetisation of Physical Protons for Douglas fir. At high values of physical proton intensity, the compressive strength of the specimens is constant. At approximately 40% of the maximum physical proton intensity, however, this changes. As the physical proton intensity diminishes, the compressive strength of the wood increases linearly.

Similar observations again were made for mahogany and redwood samples. These graphs show remarkable correlations between the NMR parameters and the compression tests. The graphs are directly analogous to correlations between moisture content and strength, which have been well-documented. Consequently, it is obvious that direct relationships exist between NMR parameters and the strength of wood. Specifically, the intensity of magnetisation of the physical protons is directly related to the ultimate compressive stress of a wood specimen. Likewise, the ratio of the physical to chemical proton signal intensity is also directly related to the ultimate compressive stress of a wood specimen.

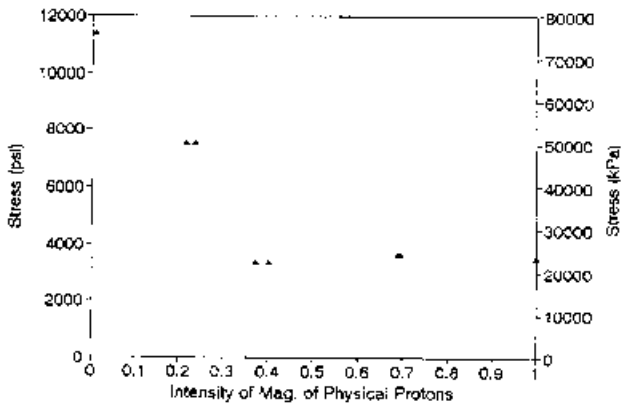

Figure 12. Intensity of magnetisation of physical protons *vs* ultimate compressive stress (Douglas fir)

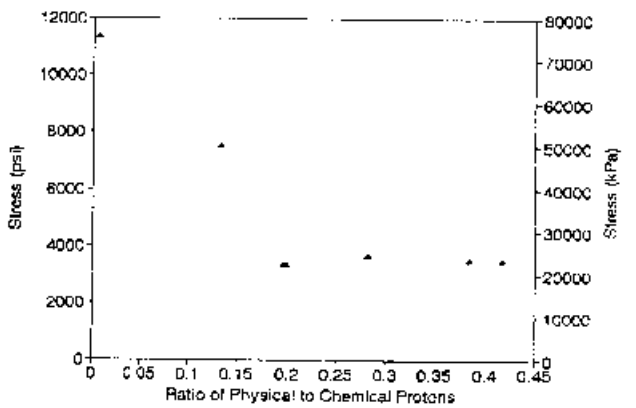

Figure 13. Ratio of physical to chemical protons *vs* ultimate compressive stress (Douglas fir)

Another significant result of the NMR tests is the abrupt change in ultimate strength that occurs as the physical proton signal diminishes. In the destructive tests, the presence of a 'fibre saturation point' was mentioned, which corresponds to the moisture content at which the mechanical properties of wood begin to be affected. Above this point, the wood strength is constant. The NMR parameters very distinctly show this same abrupt change in wood strength. Therefore, the NMR response can be calibrated to predict the strength of wood with a reasonable degree of accuracy.

These preliminary results are very promising. Since NMR spectroscopy is a non invasive procedure, there is great potential to be able to measure the strength of wood specimens without damaging the integrity of the wood. It is obvious that further testing should be done in order to develop specific procedures that may be followed in order to actually measure the strength of in place wood members by using a portable NMR spectrometer.

Reproducibility of results

A correlation between parameters is only valid if the correlation is proven to exist under repeated trials. In other words, the results of any experiment must be shown to be reproducible in order for those results to be valid. For this experiment, the reproducibility of results was examined in several ways.

The reproducibility of the NMR results was examined in two ways. First, during each round of testing, the mahogany sample MH2 was analysed twice. Before each run, the frequency and phase of the spectrometer were adjusted to be sure that the system was in resonance. Therefore, the results of each experiment represented two separate and totally independent analyses of the NMR parameters. The two consecutive runs were compared and the average change in the two measured values of each NMR parameter was calculated. The results of this comparison are shown in Table 1.

Table 1. Comparison of NMR parameters in consecutive tests

	PARAMETER	AVERAGE PERCENTAGE CHANGE IN PARAMETER
BI-LINEAR FIT TO FID	Physical (0)	2.13%
	Chemical (0)	1.40%
	Ratio	0.81%
ECHO	T_2(fast)	14.4%
	T_2(med)	19.5%
	T_2(slow)	-

The bi-linear fit to the FID appears to yield extremely reliable results. The percentage change of the parameters from one test to the next averaged from only a couple percent to fractions of a percent. This high level of precision illustrates the tremendous promise that NMR spectroscopy has as an analytical technique. The echo pulse yielded only moderately reliable results, as discussed earlier.

The second means of gauging the reproducibility of the NMR results was to test two identical specimens of each species under the same experimental conditions. The specimens were tested at almost the exact same moisture content. The reproducibility of the results may be examined by referring to Figures 8 through 10, which plot the NMR parameters *versus* moisture content. The regression analysis of each graph yields the slope of the regression line, which is termed 'X coefficient'. This slope expresses the relationship between the NMR parameter and the moisture content of the sample. For two identical specimens of the same species, this relationship should not

vary significantly if the correlation is valid. For each parameter that was measured the X coefficients of the two identical samples were compared. The percentage change of the X coefficient between the two samples was then calculated. The results of this comparison are shown in Table 2.

Table 2. Comparison of the X coefficient (or slope) of the linear regression of two identical wood samples

	PARAMETER	AVERAGE VALUE	PERCENTAGE CHANGE
DOUGLAS FIR	P(lin)	0.01974	0.99%
	C(lin)	0.01100	6.04%
	R(lin)	0.008608	1.19%
MAHOGANY	P(lin)	0.01611	2.22%
	C(lin)	0.009621	1.22%
	R(lin)	0.010386	0.24%
REDWOOD	P(lin)	0.01409	9.31%
	C(lin)	0.007595	3.80%
	R(lin)	0.009800	3.82%

The NMR parameters have been abbreviated as follows: the intensity of magnetisation of physical protons as calculated by a bi-linear fit to the FID is termed P(lin). The remaining abbreviations are similar. Also included in the Table are the average values of the NMR parameters for each species.

The reproducibility of most of the parameters is very good. For example, P(lin), C(lin) and R(lin) all exhibit extremely stable results. The percentage change in the value of the X coefficient of the regressions of only one or two percent shows that the correlation between these parameters is viable and reproducible.

It should be noted that the X coefficient for the regression between the physical proton intensity and the moisture content appears to be noticeably different for the three species (see P(lin) in the Table). Although more tests need to be run in order to prove this result, it does represent a possible correlation between an NMR parameter and species type.

Summary and conclusions

The feasibility of using NMR spectroscopy to measure the mechanical properties of wood non-destructively has been investigated. Specifically, samples of Douglas fir, mahogany, and redwood were tested in compression and a correlation was sought for with parameters calculated by NMR testing methods.

The non-destructive testing was carried out on a small, portable NMR spectrometer. Conventional spectrometers, due to their size and expense, are not suited for use in field operations. Samples of Douglas fir, mahogany, and redwood were tested at moisture contents very close to that used in the destructive tests. From the FID and the echo envelope curves, the NMR parameters were calculated.

Two NMR parameters were found to correlate well with the moisture content of the wood. The intensity of magnetisation of the physical protons (free water), and the ratio of the physical to chemical protons both vary directly with the moisture content.

A correlation was sought for between the NMR parameters and the destructive properties of the wood samples. It is thought that this represents the first attempt to link NMR properties to the strength of wood. The compressive strength of the wood was plotted *versus* the intensity of magnetisation of the physical protons and *versus* the ratio of physical to chemical protons. The correlation between the parameters is excellent. The presence of 'fibre saturation point' at which the strength of the wood begins to increase rapidly is readily apparent in the graphs.

These preliminary results are very promising. NMR spectroscopy appears to be capable of estimating the moisture content, and when calibrated, the compressive strength of a wood sample. The fact that this is a non-destructive technique makes it remarkable. Furthermore, these results were developed using a small spectrometer that could be used in the field, which is a necessity if the strength of existing structures is to be investigated. Additional research is needed to develop techniques and databases for estimating strength of wood samples in the field.

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