Application Note 01662

Microimaging Applications in Industry – Curing of Plaster Cement Studied by MRI

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Introduction

MRI is widely used in the biological and medical fields. Its application in material science has been limited mainly due to poor signal-to-noise ratio resulting from a low abundance of protons and T2* related signal loss. To demonstrate the applicability of MRI in materials research, the curing process of plaster cement was studied as a function of time. Plaster cement is widely used in the building industry as a key component of the dry wall material. Its characteristics and performance are determined by hydration and dehydration during the curing process. NMR spectroscopy and imaging techniques are valuable because they provide non-destructive methods for studying the changes in water properties inside materials (1,2). T1, T2 and diffusion are also useful NMR parameters that can be studied and correlated to the properties and performance of the plaster cement (3).

Experimental

The experimental studies were done using a 400 MHz Varian NMR System. The spectrometer was equipped with the standard microimaging hardware including a 45 mm, 100 G/cm gradient coil and a 25 mm proton RF coil. A sample of commercially available plaster cement (DAP brand) was placed in a 1 cm plastic cap with the top surface exposed and placed inside the RF coil. The T1 and T2 measurements were done using the Inversion Recovery (IR) and Carr-Purcell-Meiboom-Gill (CPMG) sequences, respectively. The imaging experiments were done using the 3-D Spin Echo (SE3D) sequence. Hard 90- and 180-degree pulses were used in the SE3D sequence to keep the echo times short and increase the signal level.

Imaging Protocol Parameters

Field of View:	20 mm
Recycle Time (TR):	600 ms
Echo Time (TE):	5.5 ms
Averages:	2
Image Resolution:	0.3 mm (read) 0.6 mm (phase)
Scan Time:	20 min

Results

The NMR spectrum of the sample showed a broad water spectrum indicating T2* related line broadening (Figure 1). This is typical of most samples used in material science, such as polymers, wood and concrete.

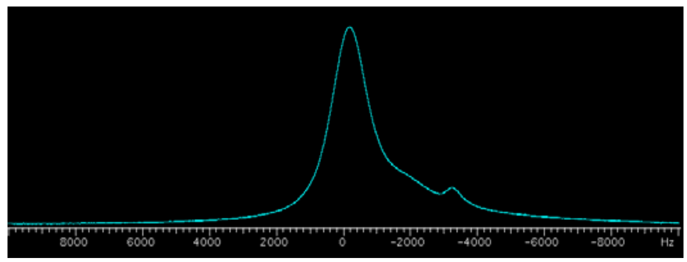

Figure 1. The proton NMR spectrum taken from a sample of dry wall plaster cement at 400 MHz.

Using the spectral analysis protocols in VnmrJ™ software, the T1 and T2 of the sample were determined to be 144 msec and 4 msec, respectively. T1IR experimental data is shown in Figure 2 and was used to measure T1 of the water signal.

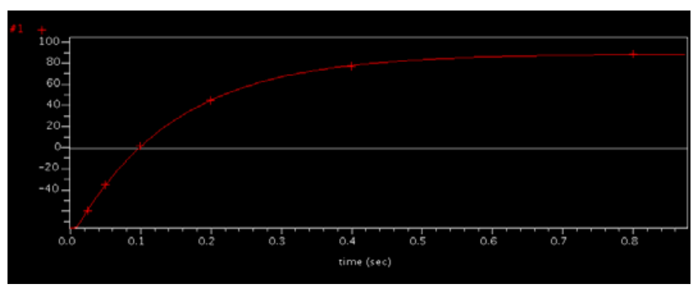

Figure 2. The T1 relaxation curve of the water component in plaster cement measured using the T1IR sequence.

Unlike the spectroscopic analysis, MRI methods provide information on the spatial distribution of water. Therefore MR images can provide a more complete picture of the changes taking place in the water at various locations within a sample. The spin-echo sequence is particularly beneficial in these applications because the 180 degree refocusing pulse helps to minimize the T2* related signal losses. Four adjacent slices taken from a 3-D image dataset are shown in Figure 3 and were taken at 1-, 24- and 96-hour intervals after sample preparation. The image intensity is directly related to the water signal and provides a convenient, non-destructive way to visualize the curing process.

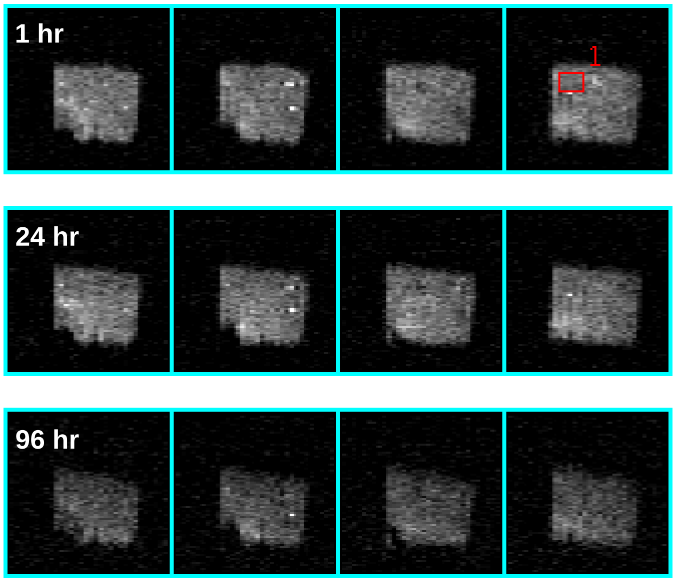

Figure 3. Each set of four images (left to right) represents adjacent slices taken from a 3-D spin-echo image dataset. The top, middle and bottom set of images (taken at 1-, 24- and 96-hr intervals, respectively) show changes during the drying process. A rectangular region of interest in slice #4 (top, right-most slice) was chosen to measure the water signal intensity at various times.

Though the change in image intensity (water signal) at various times is not obvious in Figure 3, a quantitative measurement shows the changes occurring during the curing process. Using the graphical tools in VnmrJ™ software, a rectangular region of interest was drawn in slice #4 (Figure 3) and the mean signal intensity measured at various time (Table 1).

Table 1. The change in water intensity (arbitrary units) in the selected region of interest, from slice #4 in Figure 3, as a function of time.

	1 h	2 h	4 h	22 h	28 h	96 h
Mean Signal	6285	6215	6183	5372	5212	4596

During the curing process the loss of "free" water results in hardening of the cement material. This can be seen in the disappearance of the image intensity at the top, exposed, surface of the sample in Figure 3. The images help us view inside the sample to study the uniformity of the mixture, investigate defects and monitor changes over time. During curing, a chemical reaction takes place referred to as hydration.

Initially there is considerably more water for hydration. Exposing it to air causes the hardening. During this process, evaporation must be carefully controlled because rapid loss of water will delay or prevent hydration. Therefore retaining water during the first few days is important. Monitoring and controlling the loss of water is a critical step in the curing process because it will greatly influence the strength, durability, and water repellent properties of the finished material. As we have demonstrated here, MRI can be a useful tool for monitoring, and studying the changes in water within plaster cement.

Conclusion

Generally, materials such as cement and plaster are difficult to study by MRI because of low water content and T2* related signal losses. Nevertheless, by careful selection of pulse sequences and experimental parameters, MRI can be a valuable tool, for studying the curing process of plaster cement. MRI can also provide a non-destructive visual tool for detecting defects and water distribution within a sample. NMR parameters, such as T1, T2 and diffusion coefficients, can provide information on the water properties in cement and other materials, which may help to understand their physical and mechanical properties. Varian VnmrJ software provides an easy-to-use interface for setting up and running the protocols, such as those mentioned in this article. The 2-D and 3-D image display routines provide graphical tools for visualizing and analyzing images.

References

1. "Pure Phase Encode Magnetic Resonance Imaging of Concrete Building Materials", J. J. Young, et.al. Chapter 3.4 page 285 in "NMR Imaging in Chemical Engineering" Editors S. Stapf and S. Han, Publisher Wiley-VCH.
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