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Gadolinium chloride as a contrast agent for imaging wood composite components by magnetic resonance

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Abstract

Although paramagnetic contrast agents have an established track record in medical uses of magnetic resonance imaging (MRI), only recently has a contrast agent been used for enhancing MRI images of solid wood specimens. Expanding on this concept, wood veneers were treated with a gadolinium-based contrast agent and used in a model system comprising three-ply plywood panels fabricated from two untreated veneers and one treated veneer. The limited degree of effect imparted by the contrast agent for specimens in a water-saturated state likely resulted from contrast agent losses due to leaching during the prerequisite water saturation process. Specimens were also analyzed in the air-dry state using a non-conventional MRI sequence. This allowed what appears to be the first reported visualization of earlywood and latewood bands in MR images for air-dry wood specimens. Observation of significant signal dropout for the gadolinium-treated veneer demonstrated the first successful use of a contrast agent to manipulate the signal intensity of a wood component within a composite structure. This technique shows promise for nondestructive two- and three-dimensional assessments of wood component (e.g., veneers, flakes, particles) distributions and orientations in wood composites.

Keywords: earlywood; gadolinium chloride; $GdCl_3$; ion exchange; latewood; magnetic resonance imaging (MRI); plywood.

Introduction

Magnetic resonance imaging (MRI) has been applied to a limited extent to the characterization of internal features (e.g., knots, decay) in solid wood specimens (Wang and Chang 1986; Chang et al. 1989; Dawson-Andoh et al. 2001; Bucur 2003). For conventional spin-echo MRI, the moisture content (MC) of the specimen must be above the fiber saturation point to obtain an image (Müller et al. 2001, 2002). This is because the images are essentially derived from the proton MR signals of free water (Olson et al. 1990; MacMillan et al. 2002). Protons from cell wall components (e.g., cellulose), and bound water in wood below the fiber saturation point have short spin-spin (T_2) relaxation times and so their signals have mostly decayed prior to data acquisition (Olson et al. 1990; MacMillan et al. 2002). Thus, zones in wood with low MC have relatively low signal intensities and appear as dark regions in the image; zones high in MC have higher signal intensities and appear as bright regions.

However, the use of ultrashort time echo (UTE) methods, such as single-point ramped imaging with T_1 enhancement (SPRITE; Balcom et al. 1996), allows the detection of short T_2 components. MacMillan et al. (2002) were successful in applying SPRITE to the imaging of wood samples with MC below the fiber saturation point, although anatomical features were not resolved. Recently, the SPRITE experiment was modified to diagonal SPRITE (d-SPRITE), allowing data to be sampled more rapidly and thus providing higher resolution within a shorter time frame (Protti et al. 2005).

In medical applications of MRI, paramagnetic contrast agents are often used to enhance the contrast between healthy and unhealthy tissues by altering the T_1 (spin lattice) and T_2 relaxation times of water protons in these tissues (Almén and Aspelin 1995; Tóth et al. 2002). Most contrast agents are based on chelates of gadolinium(III) (Gd^{3+}), which has the highest possible number of unpaired electrons, making it the most paramagnetic stable metal ion (Tóth et al. 2002). Although dysprosium has the highest magnetic moment of all lanthanides, its T_1 relaxivity effect is a fraction of that for Gd (Rocklage and Watson 1993; Sorensen et al. 1997). Significant shortening of T_1 relaxivity on infusion of Gd-based contrast agents results in signal enhancement during conventional T_1-weighted spin echo MRI (Sorensen et al. 1997). At this juncture it should be noted that reductions in T_1 and T_2 relaxation times are concentration-dependent and greater T_2 effects at relatively high Gd concentrations can result in a decrease in signal intensity (Almén and Aspelin 1995).

The first application of a contrast agent for imaging wood defects by MRI was reported by Eberhardt et al. (2006). Treatment of knot-containing spruce wood blocks with a gadolinium chloride ($GdCl_3$) solution, in combination with solvent pretreatment, permitted the visualization of abrupt signal losses and thus the apparent demarcation of compression wood zones around softwood knots. Expanding on this concept, it was of interest to deter-

mine if the treatment of wood composite components (e.g., veneers, flakes) with $GdCl_3$ could be useful to distinguish these components from untreated components imaged by MRI. Here we report our results from a model system based on three-ply plywood panels fabricated from two untreated veneers and another containing various levels of $GdCl_3$.

Materials and methods

Preparation and analysis of treated veneers

Southern yellow pine veneers were obtained from a local plywood plant, cut into small sections (150 mm×150 mm×3.175 mm) and sorted to remove samples with unacceptable defects (e.g., rough veneer, staining). Gadolinium(III) chloride hexahydrate (Aldrich Chemical Co., Milwaukee, WI, USA), which is readily soluble in water, was added directly to deionized water to prepare treatment solutions of 50, 5 or 0.5 mM. Selected veneers were submerged in a given treatment solution in an open vessel that was then placed in a bell jar subjected to a vacuum to facilitate solution penetration. Vacuum was applied (15 min), released (15 min), applied again (15 min) and released again, followed by further soaking (1 h). Specimens were subsequently blotted with a tissue, weighed, and dried under ambient conditions. The gadolinium content of oven-dry (103°C) veneer samples was determined by inductively coupled plasma-optical emission spectrometry (Galbraith Laboratories, Inc., Knoxville, TN, USA). The effect of water saturation on Gd retention was assessed by cutting matchstick-sized specimens (2 g) from the veneers and soaking them in deionized water (100 ml) for 3 days. A vacuum was applied periodically to facilitate water penetration. These specimens were dried under ambient conditions and then in an oven before determination of the Gd content. Samples of earlywood (EW) and latewood (LW) were obtained by carefully slicing small sections of veneer in the longitudinal direction with a razor blade. Samples of southern yellow pine compression wood and normal wood were obtained by collecting increment cores (5 mm in diameter) from neighboring trees (either straight or severely leaning) in a local experimental forest. These samples were treated with Gd and then analyzed for Gd content before and after the water saturation process described above.

Panel assembly

Air-dry veneers were assembled into three-ply plywood panels with waterproof glue (Gorilla Glue, Cincinnati, OH, USA) according to the manufacturer's directions for bonding wood. In an effort to simulate a typical plywood adhesive mix, furfural residue filler (0.75 g, FuraTex, Bates & Co, Inc., Orange, TX, USA) and hard wheat extender (0.75 g, HW-200, Bates & Co. Inc.) were added to the glue (8.5 g). As typical of a three-ply plywood panel, the center veneer was placed in a perpendicular orientation to the face veneers. First, two untreated (control) veneers were pressed together (690 kPa, 4 h) in a Carver (Menomonee, WI, USA) laboratory press at ambient temperature. After standing overnight, a Gd-treated or untreated veneer was then bonded in the same manner to each two-ply assembly. This afforded three-ply plywood panels with either a $GdCl_3$-treated or untreated veneer, bonded in the same orientation as an opposing untreated face veneer (Figure 1). Thus, direct comparisons of the face veneers could be made to assess the efficacy of Gd treatment. Specimens (30 mm×15 mm) for MRI were cut from the panels with the long axis parallel to the grain of the surface veneers. Optical images were collected on a Leica Z16 Macroscope equipped with an integrated Z stepper and a JVC KYF-753-CCD camera (Meyer Instruments, Houston, TX, USA). Images were processed using Auto Montage software (Syncroscopy, Frederick, MD, USA).

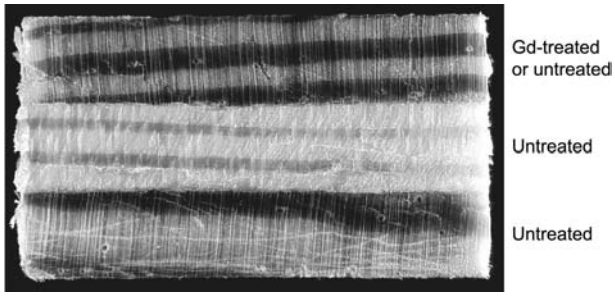

Figure 1 Optical image of a representative three-ply plywood specimen showing ply arrangements for MR imaging.

Magnetic resonance imaging

Three-ply plywood panel specimens were hydrated for conventional MRI by immersion in deionized water, with a vacuum applied to facilitate water penetration. Vacuum was applied (15 min), released (15 min), applied again (15 min) and released again, followed by further soaking of the samples overnight. Excess water was blotted off each specimen before individually wrapping them in plastic wrap. A tube of water was placed adjacent to the specimen to provide a reference for normalization of signal intensity (SI) between scans. Each specimen was then placed into a birdcage coil tuned to the ^1H frequency (25 mm inner diameter, 25 mm in length; Magnetic Resonance Laboratories, Oxford, UK) for imaging at 9.4 T in a horizontal-bore MRI scanner (Inova, Varian Inc., Palo Alto, CA, USA). Consecutive transverse MRI scans were obtained using a two-dimensional conventional spin-echo sequence: repetition time (TR), 1000 ms; echo time (TE), 10 ms; field of view (FOV), 45 mm×45 mm; matrix size, 256×128; four averages; 2-mm slices; and acquisition time, 128 min. Regions of interest covering the images of treated and untreated (control) face veneers and the reference were selected, avoiding the edges of the veneers to exclude surface water. SI values for these regions of interest were measured by ImageJ (National Institute of Health, Bethesda, MD, USA). Normalized SI values were calculated by dividing each veneer SI value by the corresponding reference SI value. Differences between normalized SI values for treated and control veneers were tested for significance based on the Kruskal-Wallis test, with a level of $P<0.05$ considered significant. If significantly different, values were compared further by means of the Dunn's multiple comparison post-test.

Two air-dry plywood specimens were imaged simultaneously for each MRI experiment with the d-SPRITE MRI sequence (Protti et al. 2005). After stacking the two specimens, they were placed together in the MRI coil and d-SPRITE MRI was performed. The following parameters were used: TR, 0.05 ms +0.12 ms=0.17 ms, where 0.05 ms is a dead time left to avoid eddy currents and 0.12 ms is the time phase (Tp, equivalent to TE); flip angle, 5°; one scan; FOV, 30 mm×30 mm×30 mm; matrix size, 100×100×11; and acquisition time, 24 min.

Results and discussion

Three-ply plywood panels were selected as a simplified model system for wood composites since the exterior veneers (one with and one without Gd treatment) could be readily compared. A sample of commercial plywood

was successfully imaged in the water-saturated state using a conventional spin-echo MRI method. Excessive swelling and adhesive failure in a water-saturated oriented strandboard specimen indicated that the selected model (i.e., plywood) was preferred. Nevertheless, it should be mentioned that water absorption was successfully monitored by MRI in a study on thickness swelling in oriented strandboard (van Houts et al. 2004, 2006).

Imaging of water-saturated specimens

A representative plywood specimen is shown in Figure 1 for comparison to the MR images. The diagram shows that the top veneer of each plywood specimen was treated with $GdCl_3$; the exception was the control, in which the top veneer was untreated. For water-saturated specimens, the MR images (Figure 2) generally show alternating layers of EW and LW within each veneer, with the former having a higher SI than the latter. This is illustrated in Figure 2a by arrows demarcating EW (light bands) and LW (dark bands).

Images obtained for the control specimen with an untreated top veneer (Figure 2a) and test specimens containing a top veneer treated with 0.5 and 5 mM $GdCl_3$ (Figure 2b,c, respectively) were quite similar. In contrast, the MR image of the specimen in which the top veneer was treated with the highest concentration of $GdCl_3$ (50 mM) showed an apparent decrease in SI for the treated veneer (Figure 2d). Comparison of the normalized SI from the 50 mM Gd-treated veneer showed it was significantly lower than that of the untreated veneer ($P < 0.05$, Table 1). Non-parametric statistical testing of the data showed no significant decrease in normalized SI values for 0.5 and 5 mM Gd-treated veneers compared to the untreated veneer; however, SI values for the Gd-treated veneers were generally lower than for the untreated veneer (Table 1).

Further examination of the series of images obtained for the specimen treated with 50 mM Gd revealed a decrease in SI for the middle veneer. This may have resulted from diffusion of unbound contrast agent, present in free form in the treated top veneer, to the middle veneer. Thus, the treated veneers in specimens before and after water saturation were analyzed for Gd content to determine if Gd in free form was being lost or transferred to neighboring veneers during the water saturation process.

Gd is conventionally considered a T_1 contrast agent. However, at the concentrations used in this study, the effect of Gd on T_2 relaxation supercedes the effects on T_1 relaxation. Preliminary work with low-field NMR was conducted to demonstrate the impact of Gd treatment on T_2 relaxation times. Measurements were carried out under conditions similar to those reported elsewhere (Eberhardt et al. 2007), and gave T_2 relaxation times of 41 and 14 ms for water-saturated solid wood (144% MC) and wood meal (154% MC) samples, respectively, by monoexponential fitting of the data. These values compare well with those (20.3–68.8 ms) reported by Meder et al. (2003) for pine wood (38–176% MC). For a sample of wood meal treated with 50 mM $GdCl_3$ (141% MC), the T_2 relaxation time was indeed shorter (7 ms).

Gadolinium content of veneers

Treated veneer samples were analyzed for their Gd content before and after water saturation (Table 1) to screen for loss of the contrast agent as a result of the water saturation process. Veneers before water saturation had Gd content ranging from 0.01% to 0.69% (w/w). Essentially identical values were obtained for water-saturated samples at the two lower Gd treatment concentrations (0.5 and 5 mM). For the highest treatment concentration (50 mM), the water saturation process resulted in a loss of approximately 50% of the originally incorporated Gd. The difference in SI between treated and untreated veneers could have been greater if not for contrast agent losses during the prerequisite water saturation process. It is also possible that some of the free Gd (i.e., not bound to the cell wall) may have diffused into the untreat-

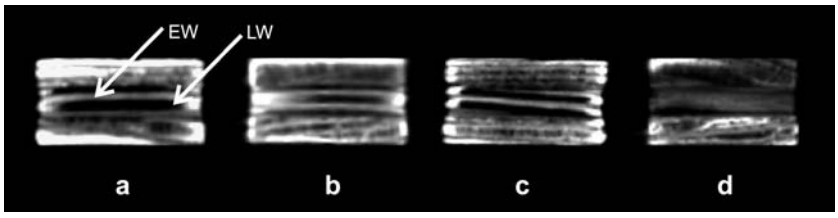

Figure 2 Spin-echo MR images of water-saturated plywood specimens with the top veneers treated with gadolinium solutions of (a) 0 mM, (b) 0.5 mM, (c) 5 mM, and (d) 50 mM (EW, earlywood; LW, latewood).

Table 1 Normalized MRI signal intensities for individual veneers (mean ± SD)* and Gd content of treated top veneers.

$GdCl_3$ treatment	Signal intensity		Gd content (%)	
	Treated top veneer	Opposing untreated bottom veneer	Before water saturation	After water saturation
0 mM	0.230 ± 0.035	0.143 ± 0.027	< 0.001	< 0.001
0.5 mM	0.101 ± 0.017	0.142 ± 0.016	0.010	0.019
5 mM	0.114 ± 0.004	0.139 ± 0.014	0.105	0.107
50 mM	0.037 ± 0.008†	0.078 ± 0.013	0.685	0.326

*Kruskal-Wallis testing was performed with post-testing by Dunn's multiple comparison if the significance level was $P < 0.05$.
†$P < 0.05$ compared to the veneer treated with 0 mM $GdCl_3$.

ed veneers, thereby further decreasing the desired effect on the images. For the veneer treated with 50 mM Gd, a sufficient amount of Gd was retained, and thus a decrease in SI was apparent for this veneer (Figure 2d). Although rapid introduction of water followed by immediate imaging may seem to be a solution to this problem, incomplete water saturation would result in dark regions (i.e., zones providing little signal) on the images owing to weak signal detection from bound protons and cell wall components that have short T_2 values.

Gadolinium content of normal and compression wood

Given the above results, we assessed variations in Gd content that could be attributed to the variability in wood (anatomy, chemistry, etc.). It is well established that woody materials have an inherent capacity to bind metal ions; studies related to the ion exchange capacity of wood have generally focused on the use of woody biomass to remove heavy metal contaminants from water (Marin and Ayele 2002; Shukla et al. 2002). To determine any differences in the retention of Gd by normal and compression woods, samples of each were treated with $GdCl_3$ and analyzed. Compression wood (CW) samples contained more Gd than normal wood (NW) (Table 2). Differences in cell wall chemistry between NW and CW have been reviewed by Timell (1986), whereas their ion exchange capacities have received little attention. The higher Gd content in CW may be related to higher lignin and/or carboxylic acid functionalities. Irrespective of the chemical constituent(s) responsible for the ion exchange capacity, the above findings are consistent with results from a previous study in which greater signal losses were observed for apparent CW zones in knot-containing wood specimens treated with $GdCl_3$ before MRI (Eberhardt et al. 2006). It should be noted that the wood veneers used in the present study did not show any obvious signs of CW formation. Comparison of NW and CW samples was simply carried out to verify that wood variability has the potential to impart variability to the Gd content.

Table 2 Gd content of selected wood samples treated with 50 mM $GdCl_3$ solution before and after subsequent water saturation treatment.

Sample	Gd content (%)	
	Before water saturation	After water saturation
Normal wood	1.07±0.27	0.37±0.04
Compression wood	1.19±0.05	0.50±0.01
Earlywood	1.59±0.13	0.41±0.00
Latewood	0.77±0.05	0.31±0.01

Imaging of air-dry specimens

To circumvent contrast agent loss during the subsequent water saturation step, we also analyzed plywood test specimens in the air-dry state using a newly developed d-SPRITE sequence (Protti et al. 2005). Plywood specimens analogous to those in Figure 2 were scanned in the air-dry state and the resulting MR images are shown in Figure 3. Application of a pulse sequence capable of detecting signals with short T_2 values clearly revealed wood features, specifically EW and LW bands of air-dry specimens, in the images. In the water-saturated state, EW bands, with their higher MC in the water-saturated state, afforded greater SI and appeared as lighter areas on the images. For air-dry specimens using the d-SPRITE sequence, the denser LW appears brighter simply because of the greater amount of material affording a signal. The observation of EW and LW bands alone was of interest since it demonstrated the feasibility of resolving wood anatomical features during MRI of air-dry (MC ~9%) specimens.

The pixilated character of the images reflects the overall low resolution owing to hardware limitations. The observation of signal dropout for the 50 mM Gd-treated veneer was of particular interest (Figure 3d); only the two untreated veneers are readily apparent for this specimen. Whereas the EW afforded a very weak signal because of its low density, the denser LW bands were also missing in this veneer through the effect of the contrast agent. Thus, we successfully used a contrast agent to manipulate the SI of a component within a wood composite structure. Improvements in this technique could lead to non-destructive two- and three-dimensional characterizations of wood component (e.g., veneers, flakes, particles) distributions and orientations in wood composites.

Gadolinium content of EW and LW

Finally, since the levels of Gd retained by CW and NW were not the same, we investigated if apparent differences in ion exchange capacity also extended to EW and LW in the veneers. Samples of EW and LW were thus treated with $GdCl_3$ and analyzed for their Gd content before and after subsequent saturation with water. Given the lower density of EW relative to LW, the higher Gd content of EW before water saturation reflects greater uptake of the Gd treatment solution (Table 2). As expected, prior to water saturation, the value for the NW sample was intermediate between the values for EW and LW

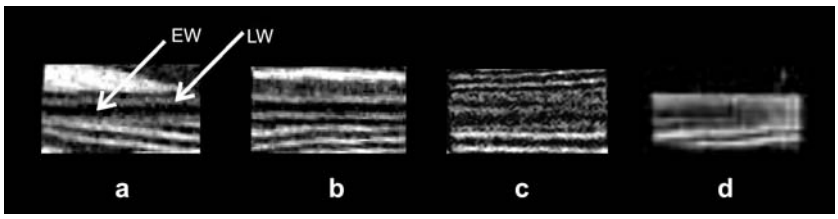

Figure 3 Diagonal SPRITE MR images of air-dried plywood specimens with the top veneers treated with gadolinium solutions of (a) 0 mM, (b) 0.5 mM, (c) 5 mM, and (d) 50 mM (EW, earlywood; LW, latewood).

samples. Likewise, the same relationship existed for samples subjected to the water saturation process. The greater loss of Gd upon water saturation for EW relative to LW (Table 2) indicates that a greater proportion of Gd present in EW before water saturation was in a freely soluble form. The lower inorganic content of pine LW relative to EW (Fengel and Wegener 1984) may thus be associated with differences in their ion exchange capacity. Results presented in Table 2 for the Gd content after water saturation are also indicative of a higher ion exchange capacity for pine EW, manifest in higher retention of Gd. The somewhat higher Gd content accompanying the greater volume of water in EW could explain, in part, the poor image contrast between EW and LW in specimens when imaged in the water-saturated state. For specimens analyzed in the air-dry state, a greater amount of Gd in EW would only enhance image contrast between EW and LW given the lower signal for EW relative to LW.

Conclusions

The limited effect of the contrast agent on images of water-saturated specimens likely resulted from leaching of the agent during the water saturation process. This problem was circumvented by analyzing specimens in the air-dry state when performing MRI by d-SPRITE. Wood features, specifically EW and LW bands of the air-dry specimens, were also readily apparent using the ultrashort TE pulse sequence. Signal dropout for the Gd-treated veneer showed that contrast agents could be useful in manipulating the SI of a wood component within a composite structure.

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