Potential of near infrared spectroscopy to quantify boron concentration in treated wood

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Abstract
Near infrared spectroscopy (NIR) coupled with multivariate statistical methods was assessed as a possible method for determining quantitatively boron-based preservative concentration in treated wood. It was found that a NIR-based model could successfully predict the borate concentration of treated pine sapwood cubes, suggesting that it may provide the basis for a rapid, easy-to-operate and portable analytical tool.

Borates have been successfully used as wood preservatives and pest-control products for many decades. They are useful because of their broad spectrum efficacy against all wood-destroying organisms including termites, wood-destroying beetles, and decay fungi, with their performance being combined with low mammalian toxicity and a low environmental impact. Borates are water soluble and are typically applied in industrial applications using vacuum pressure techniques or in remedial treatments by topical application with subsequent diffusion into the treated materials. As with all preservative systems, efficacy is dependant upon reaching a preservative concentration in the treated wood that is above the toxic threshold of the target pest(s), and thus it is important to be able to accurately determine preservative concentration. This is especially important with wood where, even with proper treatment procedures, concentrations can vary plus or minus 50 percent because of the natural variability of the wood. For in situ applications, preservative quantification is important to ensure no applicator error.

Existing methods for determining borate preservative content of wood are either only qualitative or require elaborate analytical techniques. Qualitative methods include the use of surface-applied colorimetric reagents such as catechol violet (available commercially as borate indicator test kits) or using the curcumin method described by Smith and Williams (1969) and in AWPA Standard A3-05 (AWPA 2005b). For quantitative analysis, only destructive methods can be used. Wood samples must be reflux extracted and the resultant solution analyzed by colorimetric titration using a spectrophotometer (AWPA A2-05, AWPA 2005a) or by potentiometric titration, or by other methods such as inductively coupled plasma emission spectroscopy (ICP AES) or ICP mass spectroscopy (AWPA 2000). These techniques are relatively time consuming and can have compatibility or contamination issues resulting from sample preparation.

An additional possible method for assessing quantitative boron concentration in treated wood is near infrared spectroscopy (NIR) coupled with multivariate statistical analysis. NIR is rapid and easily applied to wood, and a number of studies have suggested that NIR spectra can be related to a variety of wood properties (reviewed in So et al. 2004b). NIR-based methods also have been shown to have the potential to quantify the concentrations of copper, chromium and arsenic in preservative-treated wood and to distinguish between various preservative formulations (So et al. 2004a). Because the reflectance of NIR energy off wood can be influenced by the presence of borate (Feldhoff et al. 1998), it is possible that NIR spectroscopy combined with multivariate analytical techniques could be used to quantifiably detect the presence of boron-based preservatives in treated wood. The objective of this study was to assess the ability of NIR to predict the concentration of borate in vacuum-treated pine sapwood cubes.

Materials and methods
Sets of five randomly selected cubes (25 mm), cut from a single southern pine (Pinus spp.) sapwood board, were placed in an aqueous solution containing approximately 0, 0.2, 0.5, 2, 5 or 10 percent disodium octaborate tetrahydrate (DOT, available commercially as Cellu-Treat or Tim-bor Professional from Nisus Corporation, Rockford, Tennessee). A vacuum
was applied to the submerged cubes and held for 30 minutes. After the vacuum was released the blocks were kept submerged in the treating solution for 8 days under ambient conditions in the laboratory to allow ample time for diffusion of borate throughout the block. The blocks were removed from the solution, dried in an oven at 60°C overnight, exposed to ambient laboratory conditions for 7 days, cut in half (cross section) using a band saw and the cut face scanned with a NIR spectrometer as described below. After the NIR measurements, the wood pieces were analyzed for borate content using reflux extraction in water for 1 hour and subsequent potentiometric titration of the resulting solution using mannitol (AWPA A2-05).

The NIR measurements were made with an Analytical Spectral Devices (ASD) Field Spectrometer at wavelengths between 500 to 2400 nm. A reflectance contact probe housing a high-intensity light source and a fiber optic was used to collect the reflectance spectra. A piece of Spectralon® was used as a white reference material. For each NIR spot (0.75 inches diameter), 10 scans were collected and averaged (mean) into a single spectrum. Two separate NIR spectra were collected for each wood sample. The reflectance spectra were converted to absorbance spectra. Spectra from the same samples were averaged. The data set was further reduced by averaging the spectra that were collected at 1 nm intervals, to a spectral data set at 5 nm intervals. No further transformations (normalization, multiplicative scatter correction) of the data were made. A partial least squares (PLS) calibration model was constructed using the data from 20 randomly selected samples (calibration set) to relate the NIR spectra (from 1000 to 2200 nm) to the borate concentration in the wood samples. The calibration model chosen used six principal components, and two outliers identified by the software were excluded. The calibration model was then used to predict the borate content of the 10 remaining samples (validation set) based on their spectral data. One outlier identified by the software was excluded.

**Results and discussion**

Measured borate concentration in the wood cubes ranged from 0.01 to 15.0 percent DOT. The calibration model was able to provide a good correlation ($r^2 = 0.96$) between the measured and predicted borate content of the samples (Fig. 1). The slope of the regression was 0.96 and the root mean squared error of the calibration (RMSEC) was 0.92 percent. The calibration model provided reasonable predictions of the borate content of the validation sample set (root mean squared error of prediction [RMSEP] = 2.12 percent, $r^2 = 0.86$; Figure 1).

**Figure 1.** — Actual (“measured”) borate content (%DOT) of treated wood cubes, as measured using AWPA standard method, vs. boron content predicted using NIR spectra. The solid circles (“calibration”) indicate samples used in building the calibration model. The open circles (“prediction”) are samples whose borate content was predicted using the calibration model. The “prediction” samples were excluded when developing the calibration model.

These data suggest that NIR has the potential to quantify borate concentration in treated wood. Development work is underway to build robust predictive models and to determine the possible influence of variables such as wood species, preservative formulation, and MC.

**Literature cited**


