

Advances in Analytical Techniques for Treated Wood

**Lehong Jin, Paul Walcheski, Andy Zahora,
Christoph Schauwecker and Alan Preston**

Viance LLC

200 E Woodlawn Road, Suite 350

Charlotte, NC 28217, USA

Abstract:

This paper presents information on the application of analytical techniques which have not previously used by the wood protection industry. Examples of using X-ray Fluorescence (XRF) Analytical Microscopy technology are provided in terms of studying wood preservative components distribution and penetration patterns, qualification and comparable quantification. The advantages of this non-destructive technique for chemical distribution research, chemical treatment identification, treated wood quality control, and in service monitoring are illustrated and discussed. The potential use of other analytical tools, such as portable color meters and contact angle analyzers for on-site monitoring of treated wood performance are also presented.

Key words:

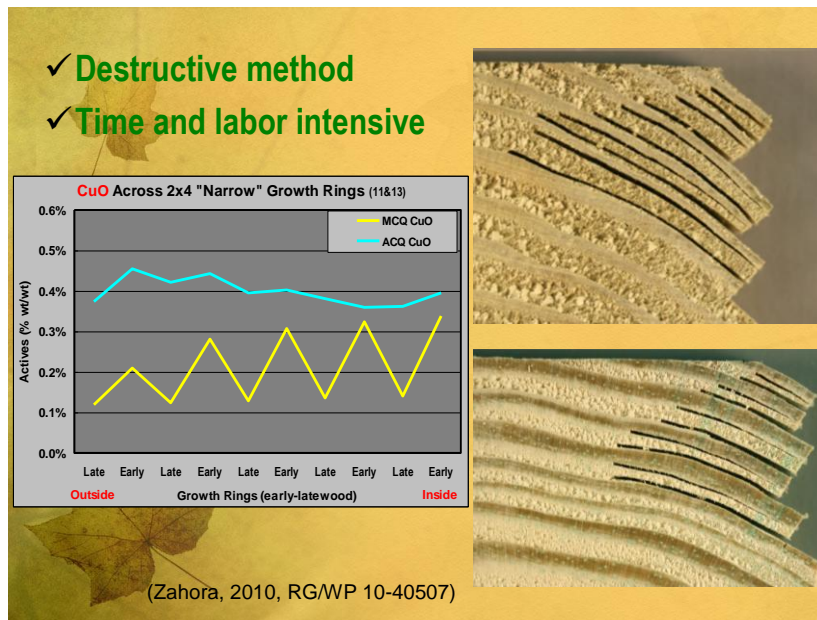
Non-destructive quality control methods, XRF Analytical Microscopy, contact angle analyzer, color meter, chemical penetration and distribution, copper based preservatives, ACQ, CA-C, Particulate copper, on site analysis, in service monitoring, water repellency, dimensional stability, color stability

Introduction

In the past two decades, the treated wood industry has undergone changes surpassing those of the previous fifty years. The traditional waterborne preservatives such as CCA have been restricted to industrial applications in most countries. Copper-based non-arsenic and non-chromium wood preservatives, such as ACQ and CuAz, have been developed and used extensively during this period, while recently, formulations such as copper-based particulate dispersion products, and carbon-based organic fungicide/termiticide formulations, as well as various forms of modified wood are increasingly being introduced into various markets. These newer systems are multi-component and are often used at lower retentions than in previous times. In some cases, though the formulations may have the same elemental components used in earlier products, the properties of these systems may differ in regards to formulation characteristics and the physical/chemical form of the components. These differences can impact the treating characteristics of these formulations such as in regard to penetration and gradient patterns.

Using color indicators for penetration evaluation is one of the key criteria currently used for quality control for treated wood. Typical copper color indicators used are Chrome Azurol (AWPA A3-2), Rubeanic acid (AWPA A3-8) and PAN Indicator (AWPA A3-14) for detecting copper penetration. Curcumin has been a common boron color indicator (AWPA A3-17). The detection limits for copper is in the 25-60ppm range depending which indicator is used while the Curcumin indicator provides around 0.8kg/m³ of B₂O₃ detection limit in wood. These color indicators provide basic information on whether the key preservative component(s) are present in the inner core area or entire cross-section of the treated wood, but may not reveal or differentiate the absolute treatment parameters, such as chemical micro-distribution or gradients.

Current industry standards require chemical core assay analyses to determine retention levels in the treated wood assay zone. In a recent paper (Zahora, IRG/WP 10-40507), Zahora has presented data from a study on the chemical distribution of copper-based preservatives across the treated zone within earlywood and latewood areas. The figure below shows the chemical analysis method used in that study which clearly demonstrated the difficulty and complexity of conducting such research for understanding the chemical distribution patterns or to provide verification of the treatments. In general, zonal assays such as used in that study are very labor intensive as the samples have to be cut and ground before running copper or other component analyses.



While Zahora's study shows marked differences between formulations in regards to distribution patterns with the treated wood, it also suggests that for both future research and quality control aspects in a changing world of wood protection there is a need for quick and preferably non-destructive tools to provide information on chemical penetration and distribution patterns, differentiate between treated wood types in service and potentially as a forensic tool for failure analyses, if and when needed.

The X-ray fluorescence (XRF) Analytical Microscope technology has been a powerful analytical tool developed mainly for forensic analysis for criminal investigation by law enforcement agencies. The technology combines optical visualization with high performance elemental analysis and imaging. Micro-XRF element imaging and analysis has become faster with better resolution by the improvement on various sizes of capillary X-ray guide tubes and its application has been extended to many other fields (<http://www.particletechnology.com/content/89/xrf-analyzer>). The range of measurable sample area extends from a minimum diameter of 10 μ m to a maximum area of 100mm X 100mm which allows macro analysis for a general survey of a wide area such as cross section of treated wood, to the characterization of a specific micro area around 10 μ m in size. This technology offers many advantages including little or no sample preparation, ease of use, significantly reduced measurement times and the ability to more accurately characterize complex samples - for elemental identification, concentration and visual image of solids, liquids and powders (Horiba 2010).

In this paper, the initial study results and potential applications of using X-ray Fluorescence (XRF) Analytical Microscopy technology for treated wood analysis are reported. Some other portable technologies for on site analysis and performance monitor are also discussed.

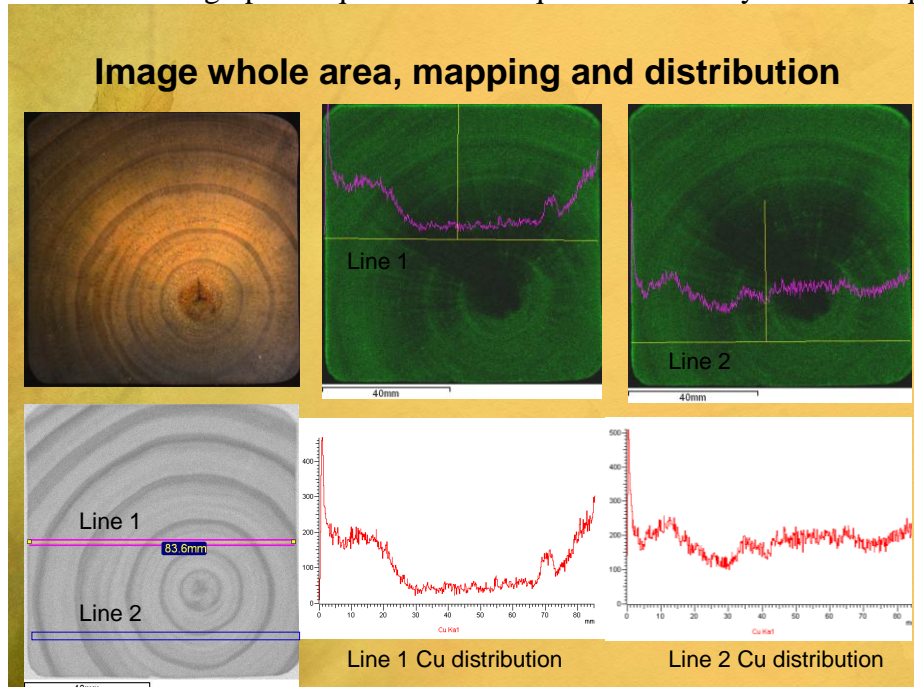
Experiments and Results

XRF Analytical Microscopy:

The instrument used for the XRF Analytical Microscopy analysis in this report is the Horiba XGT-7000 system with dual X-ray guide tubes. Several examples of the tests performed to evaluate the applicability of the analytical method for treated wood samples are presented and summarized in the next several sections. These examples illustrate the use of this technology for cross-section elemental mapping and imaging, corresponding transmission image, single or multiple lines elemental analysis of selected areas, point and multi-points elemental analysis, and multi-elemental analysis and imaging.

Example 1:

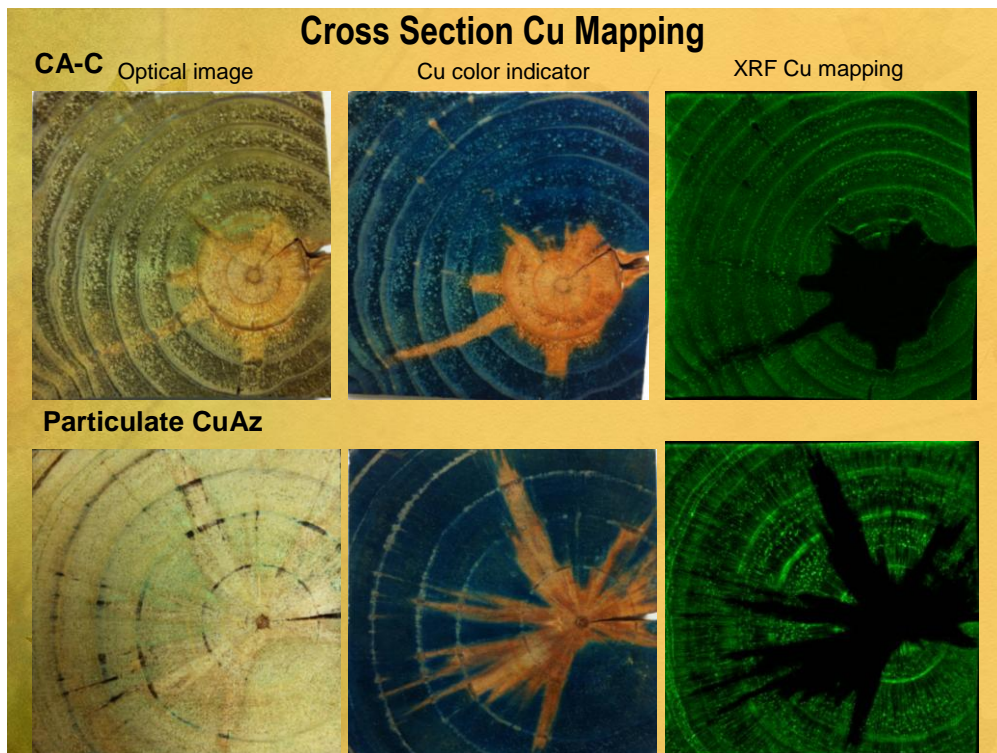
In this example, a not-fully-penetrated 4x4 post section with ACQ treatment has been examined to illustrate elemental imaging techniques. The cross-section was mapped with XRF Analytical Microscopy for optical image, transmission image and Cu elemental image as shown below. The elemental image map provides whether the chemical penetration is uniform or not, the color intensity represents the element counts at the individual points. In this case, the brighter the green, the higher the copper counts are. The dark area represents very low copper concentration. The copper counts data are available for further quantification. As indicated in the transmission image, the instrument also allows further point and line analysis. In this case, two different position lines were drawn on the same copper distribution and transmission maps, the copper distribution along the lines can be displayed in the map with the heights from the base line as indication of the concentration on copper at corresponding points. The line distribution data can also be extracted as the line graph for qualitative and quantitative analysis and comparisons.



The line and point analysis can be performed rapidly for any locations on the map. When an element mapping is generated using this technology, not just the element(s) of interesting are recorded, any element(s) presented in the substrate material will be recorded and stored in the image data file. The availability of such information allows further “mine” the data if there are such needs to determine elemental combination or contamination, metal alloy compositions and so on.

Example 2

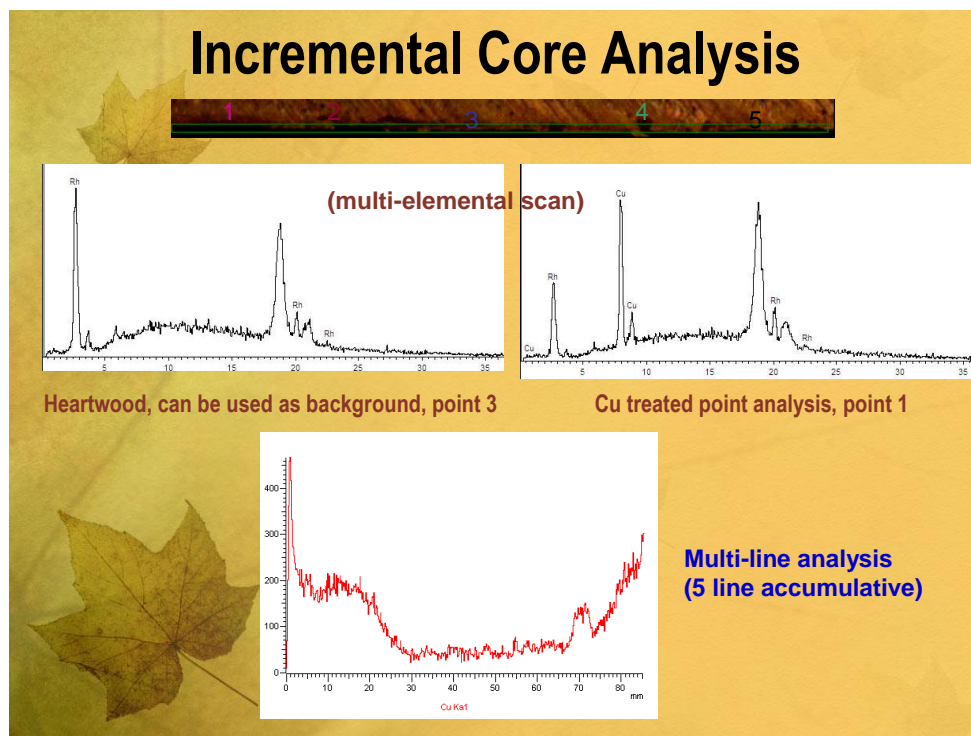
In this example, two 4x4 cross sections, one treated with soluble copper based preservative CA-C and the other treated with particulate CuAz are studied. Both cross-section samples have sapwood with copper fully penetrated based on spraying Chrome Azurol color indicator. The full cross-section copper elemental mapping was carried out the same as the example 1 and the results are shown below.



The mapping data suggest that though conventional color indicator shows full copper penetration in the sapwood areas, the details of penetration patterns can not be revealed. It is interesting to observe that copper in the soluble copper based preservative treated wood distributes in the wood in a more smooth and continuous manner while the particulate copper treatment behaves differently with a more streaky distribution. This phenomenon has been repeatedly observed in various samples. Further more detailed studies are needed to understand the possible causes for this treatment characteristic difference between these copper based preservatives.

Example 3

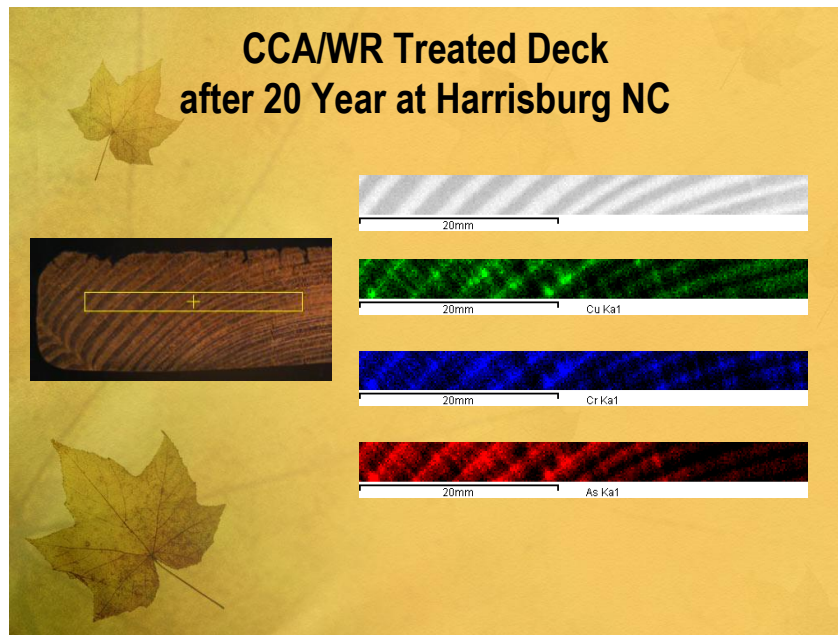
In this example, the potential use of this technology for on-site quality control or in-service monitoring of preservative penetration and retention levels with increment core scanning has been studied. A core taking from a 4x4 treated lumber (through entire cross-section with heartwood in the middle region) was used for multi-point and multi-line scanning analysis. A very fast multi-point analysis can provide chemical presence information and a quick indication of relative concentrations along the core. This can be achieved by using multi-elemental analysis for a single point. In wood treatment practice, the element of interest has been pre-determined, in this case, it is copper; a fast multi-line analysis (scan) for copper can be performed. That would provide an accumulative relative copper concentration across the core. The figure below showed results of these two applications.



By comparing background elemental spectrum (in this case the heartwood area, point 3), the spectrum for point 1 clearly demonstrated the copper was the key element at that point of the sample. A 5 line accumulative scan for this core showed very little copper in the heartwood area (30mm-65mm) and higher copper concentration was observed in the sapwood zone (0-30mm and 65-85mm) with the copper distribution gradient from high to low from outside to inside the lumber.

Example 4

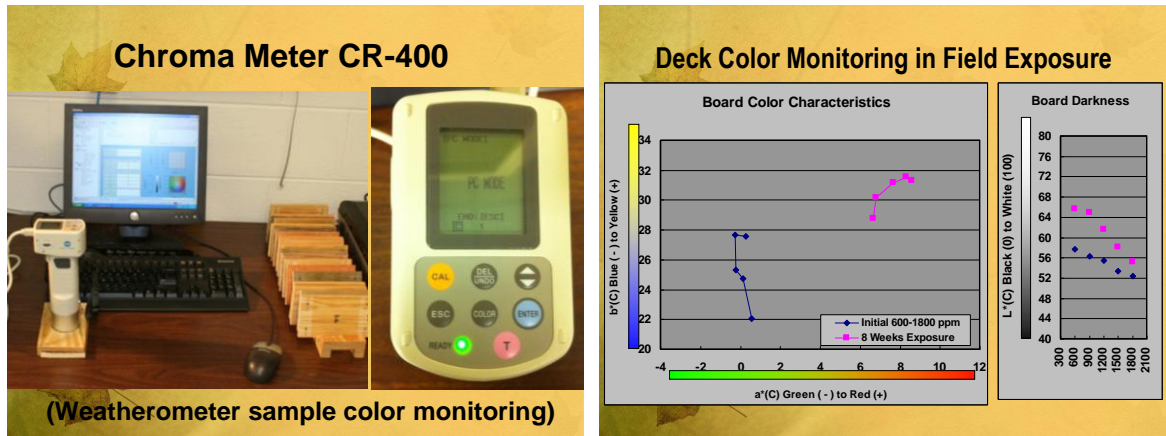
This example studied a CCA/WR treated deck that had been exposed in Harrisburg NC for more than 20 years. In this case a quick area mapping analysis was performed from a cross-section cut from one of the deck boards to visualize the three active ingredient components. The distribution maps for three elements, Cu, Cr and As were shown below. The map clearly showed that the three elements are all present in a similar manner in the treated board after 20 years' outdoor exposure.



Portable Colorimeter for monitoring color change of treated wood

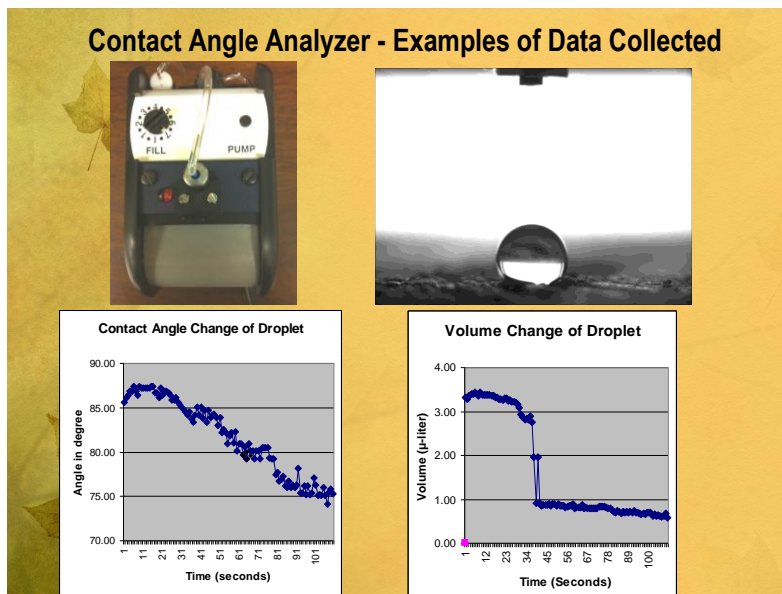
Wood that has been treated with organic preservative systems is usually colorless. With the increasing use of such preservative systems, colorants have been used in certain applications. These colorants can serve two useful purposes, one as a marker for the treated wood and the other to potentially extend the wood surface resistance to UV degradation in service. Therefore, there is a need to monitor the color change in order to assess the effectiveness of colorant.

In our experiment, A ChromaMeter CR400 has been used for assessments of color changes with time for the treated wood. This colorimeter is portable and with a built in computer system so that data can be recorded in the field (Stand mode) and then downloaded later for data processing. If it is used in a laboratory setting as shown in the figure below, it can be directly linked to a PC (PC mode) for instant recording. The data collected either in a laboratory or application sites can be presented in various color scale graphs for comparison and monitoring. The figures below showed examples of using the instrument for evaluation of Weatherometer exposed samples, and for data collected from a field deck exposure.



Portable Contact Angle Analyzer

With the increasing use of penetrating water repellents and moisture control stabilizer treatments, and hydrophobic surface additives, different types of treated wood in service can have very different surface moisture control profiles. One of the measurements to characterize these various profiles is to determine the surface water droplet properties. Portable contact angle analyzer can provide such evaluations, and modern systems are both easy to use and can provide very reproducible results. The instrument used in this study provides droplet size selection and an automated pump to control the consistency of droplet size for testing. During the measurement, a video records the droplet dispersion profile and collects the corresponding data. The video and data can then be downloaded for processing. The output provides droplet height, baseline, volume and contact angle changes with time. These data also allow for calculation of surface tension and changes. The figure below shows instrument and example of such measurement data.



Conclusions

New analytical techniques are available and applicable to our industry in meeting the challenges of adapting to new wood protection agents, technologies and treatment practices.

XRF Analytical Microscope technology can serve as a powerful research tool for determination of chemical penetration, gradients and distribution, as well as for new product development. This technique also offers potential as a non-destructive and fast evaluation tool for identification and quality of treated materials as well as a forensic tool for investigation of service performance issues.

With increasing use of surfactants and dispersion agents for new formulations, as well as value-additives for treatments, analytical evaluation methods for the effect of these components on treated wood performance need to be user friendly and field applicable. The examples of such techniques discussed here for surface hydrophobic characterization and color evaluation could provide valuable in-service data for the performance and improvement of wood protection systems.

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