

TEN YEARS ON – A REVIEW OF THE NSERC-INDUSTRIAL RESEARCH CHAIR IN WOOD PRESERVATION AT UBC.

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1. Wood pole research

The initial research direction of the Chair was driven by the student resource available and focused on the effect of service life on the preservative distribution in chromated copper arsenate (CCA) and pentachlorophenol (PCP) treated poles (Ruddick et al. 1991; Ruddick 1991a, 1991b). The project examined the pole hardness and chemical distribution, in CCA and PCP treated poles, that had been in service from a few years to almost 50 years. This data has subsequently been used by other research groups, during development of models for the loss of preservative from utility poles, as well as for estimating the residual preservative which will need to be removed during recycling of treated pole waste (Ruddick, 1990). The analysis of the CCA-treated wood showed clear trends of decreasing relative percent chromium and increasing relative percent arsenic, with increasing distance from the pole surface. The data also provided evidence of minimal loss of preservative during long term exposure of CCA-treated wood. More recently the loss of preservative from ammoniacal copper arsenate (ACA) treated poles to soil has been examined, (Ruddick and Knecht, 1997). The movement of copper and arsenic through the soil was profiled. This project is ongoing in collaboration with industry.

2. Preservative fixation

The fixation chemistry of CCA was identified during development of the Chair as a key research area. Initial research identified the important role of moisture during the fixation process, and suggested that a minimum surface moisture content of ca. 10% was required to ensure that the fixation process proceeds completely (Avramidis and Ruddick, 1989). Heating under drying conditions or at low temperatures extended the fixation time significantly.

A program was developed to use x-ray photoelectron spectroscopy (XPS) and electron spin resonance (ESR) spectroscopy to examine the changes in the oxidation states of chromium, copper and arsenic during the fixation reaction. In order to provide a sound scientific base for interpretation of data, the roles of extractive migration and blade contamination during sample preparation for XPS, were examined (Yamamoto and Ruddick, 1993). Both were found to have a major influence on the spectral properties observed. The migration of extractive to the sample surface caused an enrichment of the C1 (carbon bonded to other carbon and/or hydrogen), while the oil coating on new blades affected the O/C ratio. Subsequent studies (Ruddick et al., 1993) confirmed the role of extractives in enhancing the C1 component of the XPS spectrum. Examination of the XPS spectra suggested that decarboxylation of the wood occurred during the fixation of CCA. The fixed CCA-treated wood showed higher water repellency, particularly if this was coupled with a movement of extractives to the surface and reaction of these

extractives with the CCA. Further studies of the CCA-fixation reaction demonstrated that the fixation process involved the formation of chromium(V) which had not been shown previously as an intermediate during the fixation process (Ruddick et al., 1994). The chromium(V) species was found to be very stable in dry wood, although under conditions of moist heat it was quickly converted to either chromium(VI) or chromium(III). Predictably, the studies also clearly demonstrated that oven drying of CCA-treated wood lead to an increased loss of CCA components. Steam fixation appeared to have the greatest initial effect on chromium and copper losses, which were markedly reduced. Subsequently, the chromium leaching leveled off, while that of arsenic continued to decrease.

While the characterization of the fixation reactions in CCA-treated wood remains an important component of the research program conducted by the NSERC/Industrial Chair, the recent activity has focussed on the impact of alternative fixation processes on the fixation chemistry of CCA. A new project has been initiated to investigate the influence of radio frequency (RF) heating of treated wood, on the CCA-fixation reaction. This project was stimulated by industrial interest in the use of RF heat to precondition round wood for treatment. RF heating has many inherent advantages and current research is focussed on the RF conditioning, sterilization and fixation of CCA-treated round-wood. Recent studies have confirmed the fixation of CCA-treated hemlock lumber by RF heat (Avramidis and Ruddick, 1996) and studies in progress will shortly report on the fixation in western redcedar and Douglas-fir pole sections (Fang and Ruddick, 1999). Parallel XPS and ESR studies to examine the effects of RF heating on the fixation chemistry are planned.

While the chemistry of the CCA-fixation reactions remains an important part of the research program of my group, it became clear some time ago that the long term future of CCA use in Canada and North America was no longer secure. Arsenic and chromium are both listed on the Priority I Substances List, by Environment Canada, as chemicals the use of which it is desirable to reduce. Thus towards the end of the first phase of the NSERC/Industrial Chair a strategic decision was made to implement a research program designed to develop an understanding of the fixation chemistry of ammoniacal and amine copper based preservatives. This research lead to the identification of many factors that can affect the fixation of ammoniacal copper treated wood. For example, it was shown that the application of heat enhanced loss of copper from treated wood, (Ruddick, 1992a), while the use of conditions which promoted formation of amine-copper complexes in wood enhanced resistance to leaching. The loss of arsenic in ACA-treated wood was also demonstrated, and short-term experiments appeared to show a stable relationship of copper to arsenic of 1.5:1.

At this time the industry was concerned over the development of protocols which could identify the degree of fixation of preservatives at the treating plants. A fixation task force of the American Wood Preservers Association using data, some of which was developed by the NSERC/Industrial Chair, had shown that chromotropic acid was an acceptable rapid screening test. However, quickly it became clear that similar tests were needed for ammoniacal copper systems. The NSERC/Industrial Chair developed three simple testing

strategies, which could be used at a treating plant. These have been forwarded to the Canadian Standards Association and the American Wood Preservers Association for their consideration. The first is based upon the level of copper leached from small cores using ultrasonic leaching. The process can be done quickly, in less than one hour. The resulting leachate can be analyzed using a simple spectrophotometer or using a more precise atomic absorption spectrometer. A third strategy is to monitor the pH of the leachate. While this is able to identify those wood sample which have significant unfixed copper, when the fixation process is nearing the completion, the changes in pH are very small, so that the method is less precise (Ruddick, 1992a, 1996a and b).

Understanding the fixation chemistry of ammoniacal copper systems has become a major component of the activities of the NSERC/Industrial Chair. One of the first investigations was to establish why Douglas-fir heartwood treated with ammoniacal copper systems turns almost black. Studies by the NSERC/Industrial Chair were the first to identify that a copper(II) complex with taxifolin was responsible for this dark black colour. Spectroscopic analysis of the complex revealed it to contain two copper environments, one that was a diamminecopper bonded species, while the second contained no ammonia (Ruddick and Xie, 1994).

As with the fixation of CCA, the fixation of ammoniacal copper preservatives is likely to involve replacement of protons on lignin and hemicellulose. Experiments were initiated on the reaction between ammoniacal copper solutions and vanillin, designed to mimic the abundant guaiacyl groups found in lignin. These studies lead to the identification of the first single crystal structure of a reaction between a preservative component and a model compound for a lignin (Xie et al., 1995). The structure confirmed the formation of a stable diamminecopper(II) vanillin complex with octahedral geometry. Equally important the defining of the structure allowed the correct interpretation of ESR spectra since without such information trends in the $A_{//}$ and $g_{//}$ values can not be made.

One of the first observations that suggested the total loss of ammonia leading to copper arsenate during fixation was over-simplistic, was the enhanced nitrogen retention in wood treated with ACA, (Ruddick, 1979). A concern over the enhancement was that since the nitrogen content in wood is naturally low, enhancement could increase the potential for fungal colonization. This was confirmed in a laboratory study, by Ruddick and Xie, (1995). However, even though the fungal degradation was slightly enhanced it was clear that at the expected preservative retention, control of fungal activity would still be achieved.

The research program on the fixation chemistry of basic copper preservative systems has now been expanded to include amine-copper systems. It will also examine the relative importance of the potential fixation reactions considered to occur during fixation, on the performance of the treated wood. During the first studies, the relative importance of diamminecopper formation in wood on the leachability of the copper was studied, and preliminary results reported, (Jiang and Ruddick, 1997). Crystallographic studies of single crystals of monoethanolamine-copper-vanillin have confirmed its structure and shown it to be different from that reported for diamminecopper(II) vanillin.

3. Influence of Preservative Penetration on Performance

Towards the end of the first phase of the NSERC/Industrial Chair there was considerable interest by the Canadian industry over whether the current penetration requirements in the Canadian Standard Associations standard for CCA-treated softwood lumber were greater than necessary. The NSERC/Industry Chair developed a laboratory-based protocol to examine the influence of preservative penetration (Ruddick and Doyle, 1990). This was subsequently used to evaluate the comparison of a proposed industry penetration of 5 mm with that contained in the CSA standard of 10 mm. Samples were removed from large size treated lumber samples which had been conditioned to accelerate check formation. The results clearly showed that there was no significant reduction in performance of the 5 mm treated samples (Ruddick, 1991c). This data was in agreement with data reported by Forintek Canada Corp. using samples placed in test by the author. A new standard was designed, based upon the key contribution of the NSERC/Industry Chair.

4. Accelerated Decay of CCA-treated Wood in Waterlogged Soils

During the early 1980's the Chair collaborated with Dr. Paul Morris at Forintek Canada Corp, to a research project on the factors which caused the premature failure of CCA and ACA treated wood at Westham Island. One of the first findings was the observation of a high iron uptake by the samples exposed at this test site (Ruddick and Morris, 1991). A direct result of this work was the replacement by the American Wood Preservers Association of chrome Azurol S as the reagent for detecting copper penetration in CCA and ACA treated wood after exposure. This was done since both iron and copper react to this reagent. Further research was then directed at the possible role of sulphate reducing bacteria in causing the enhanced iron uptake. The results confirmed that sulphate reducing bacteria were present in the Westham Island soil and could mobilize iron. In a specially designed laboratory mini-soil bed, iron uptakes observed at Westham Island could be replicated in approximately one month (Ruddick and Kundzewicz, 1991). Subsequent studies of the influence of iron uptake on the performance of CCA-treated wood showed that while it did not affect the decay of untreated wood, that achieved in CCA-treated wood was enhanced. However, the samples showed significant copper losses. This suggested that an alternative mechanism for the early failure may involve factors other than the iron uptake, and might include the interaction of humic acids present in soils acting as sequestering agents for the preferential loss of copper (Ruddick, 1992). Other researchers have also identified the importance of such interactions (Stephan et al., 1993; Cooper and Ung, 1992) on the loss of CCA from treated wood exposed to microbial activity under unusual conditions, for example compost formation.

5. Factors influencing the performance of quaternary ammonium compound treated wood.

The performance of alkylammonium compounds (AACs) as wood preservatives, with particular attention given to quaternary ammonium compounds (QACs) has been the focus of significant attention by the Chair. Initial research examined the fixation chemistry and showed the importance of the ion pairing reaction, with ion exchange being limited to about 10% of the QAC in the treated wood. The microdistribution of QACs in treated wood was the subject of another study and the role of lignin was clearly

shown (Doyle and Ruddick, 1994). At the same time the Chair advanced the hypothesis that early colonization by mould and staining fungi was leading to a reduced effectiveness of the QAC during exposure of treated wood. An investigation by Andrew Doyle clearly demonstrated that *Gliocladium roseum* and *Verticillium bulbillosum* were able to degrade didecyltrimethylammonium chloride (DDAC). This was confirmed by high performance liquid chromatography (HPLC) analysis of agar medium before and after fungal growth (Doyle and Ruddick, 1993). Subsequent studies by Zheng and Ruddick (1995) and Zheng (1995) have confirmed the role of mould fungi in degrading DDAC. It was found that the degrading capacity of the mould fungi was not restricted to special tolerant strains, but could be achieved by isolates which had not been exposed to QACs before, often with equal or better degrading capacities than the isolates recovered from DDAC treated lumber. Instrumental analysis of the degradation products identified decanoic acid and decanediol as possible products. Also of importance was the role of the surfactant reaction of the QAC in influencing the experimental results. In liquid culture studies the use of a silica substrate to remove the QAC from solution greatly enhanced the ability of the fungi to degrade the QAC, (Zheng and Ruddick, 1995 unpublished observations).

The interest in the degradation of QACs led to several research projects. These have a) examined the role of fungi in remediating QAC contaminated waste (Burgel et al, 1996a and b; Dubois and Ruddick, 1997) as well as b) assessed alternative QACs and other AACs as wood preservatives (Tang and Ruddick, 1994). Since much of this work has been done under contract publication of this research has been delayed to allow the sponsors to complete their assessment of economic aspects of the research. In a recent development the mechanism of the degradation of QACs by mould fungi has been the subject of an intensive research program. The formation of a by-product by the mould fungi on DDAC treated wood was confirmed and the identity of the byproduct tentatively identified by nuclear magnetic resonance spectroscopy (nmr) and Fourier Transformed InfraRed spectroscopy (FTIR) (Dubois and Ruddick, 1998).

An important staining fungus in pine is *Ophiostoma piceae*, and a collaborative study with Dr. Colette Breuil, Wood Science, UBC, was made of the factors influencing its growth in lodgepole pine heartwood (Zheng et al., 1995).

6. Accelerated weathering of DDAC treated wood.

A parallel study of the role of DDAC in weathering of treated wood was conducted and some of the findings reported (Liu and Ruddick, 1993, Liu et al., 1994). The most significant finding of the mechanism aspect of this research was that the DDAC appears to act as a catalyst and speeds up the normal weathering process. The actual mechanism does not appear to change, based upon ESR studies of the free radicals formed in the treated wood. Earlier observations confirmed the role of copper in eliminating the action of the DDAC during weathering of treated wood.

7. Preservative treatment of Parallam

The treatment of composites with CCA and ACA is important for their acceptance in biologically hazardous end-uses. The standard creating organizations require evidence of treatment and performance of treated products. Parallam is a relatively new product,

which has found use as the main structural components in buildings. It must therefore be protected where decay could be anticipated. The Chair researched the CCA distribution in treated Parallam and also examined the influence of the Parallam on the leachability of the CCA, compared to that found in solid wood, (Ruddick and Louwers, 1993). The results showed that the glue in Parallam might act to reduce copper losses from treated material. It was also shown that the penetration of CCA in Parallam was extremely high due to the relatively high void volume of the product. This enhanced CCA penetration to such a degree, that techniques such as incising did not improve it, even in Douglas-fir.

8. Remediation of PCP-treated pole waste

A major research contract project focussed on the remediation of pentachlorophenol-treated utility poles. The project examined the potential of bacteria and fungi to degrade PCP. It was found that while PCP content rapidly decreased during exposure to *Phanerochaete chrysosporium*, the chemical was simply converted to the corresponding anisole, which is even more problematic than the PCP (McBain et al., 1993). The same research did however confirm that both *Rhodococcus chlorophenolicus* and *Flavobacterium* sp. could degrade PCP in wafers prepared from waste poles containing over 10,000 ppm (McBain et al., 1995). This research also formed the basis for analytical methodology to determine PCP and dioxins in preservative treated wood (Cui and Ruddick, 1994; Cui and Ruddick 1998). As part of this program the use of supercritical fluid extraction of PCP-treated wood waste was patented together with a novel method of destroying the dioxins (Ruddick and Cui, 1995). This process is also the subject of a second patent application still in process. Consequently, none of this research has been published at the present time.

Disposal of treated wood waste remains a research interest of the Chair (Ruddick, 1995) and recent studies have examined the interaction of treated wood and the environment. Proposals on the recovery and recycling of treated wood have been developed and are being considered by industry and potential collaborators. The need for techniques designed to identify the presence of contaminants has been identified and preliminary experiments have assessed alternative techniques, (Voss *et al.*, 1994).

9. Miscellaneous projects

Collaboration by the Chair with other researchers in the Wood Science Department has expanded the range of projects undertaken on behalf of industry. The perception of retailers of treated wood products provided a valuable insight into the marketing of treated wood to the "do-it-yourself" industry sector (Cohen et al., 1992). Key findings were, the lack of customer identification with brands of treated wood, and the major concern over basic wood quality outweighed concerns over any technical aspect of treatment. It is likely that if done today such a survey would identify environmental impact as being equally important. A contracted research project, was to investigate the use of telephone book pages and waste plastic as raw materials for production of wafer like composite products. The results confirmed that composite sheets bonded with only plastic could be produced, which had bending properties comparable to particleboard (Ellis et al., 1993). However, such sheets showed high sensitivity to moisture.

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