

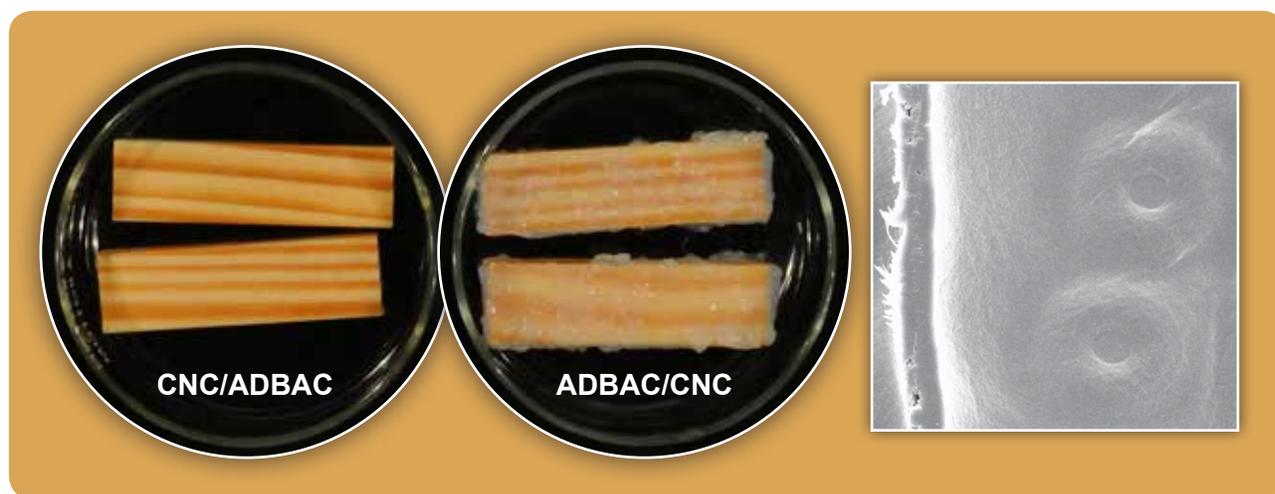


United States Department of Agriculture

Cellulose Nanocrystal Entrapment of Benzalkonium Chloride in Southern Pine

Biological, Chemical, and Physical Properties

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Forest
Service

Forest Products
Laboratory

Research Paper
FPL-RP-680

September
2015

Abstract

Protecting wood products from biodeterioration has been a dynamic area of research in the past decade with an emphasis on the development of non-arsenical wood preservatives. Naturally occurring cellulose nanomaterials that are reported to have unique chemical properties, high strength, and stiffness were evaluated for the potential to improve durability of wood either as a stand-alone treatment or in combination with a cationic biocide. In this study, benzalkonium chloride (ADBAC) was selected as a biocidal counter-cation for the sulfate ester on cellulose nanocrystals (CNC). In a two-step pressure treatment, specimens of southern pine were impregnated with CNC or ADBAC to form a CNC/ADBAC hybrid agglomerate within the wood. Cellulose nanocrystals penetrated wood in the CNC control and when CNC was the first step in a dual treatment with ADBAC. However, when ADBAC was the first step in a dual treatment with CNC or pre-mixed with CNC, the agglomerated ADBAC/CNC could not penetrate wood and gelled on the wood surface. Biological and chemical analysis of wood treated with the CNC/ADBAC hybrid revealed that CNC provided no additional resistance to fungal colonization, termite attack, or chemical leaching when agglomerated to ADBAC in wood. Analysis of modulus of elasticity, modulus of rupture, work to maximum load, and mass loss during combustion showed no change in the physical properties from the addition of CNC to wood impregnated with the CNC/ADBAC hybrid

September 2015

Clausen, Carol A.; Yang, Vina W.; Arango, Rachel A.; Hasburgh, Laura E.; Lebow, Patricia K.; Reiner, Richard S. 2015. Cellulose nanocrystal entrapment of benzalkonium chloride in southern pine: Biological, chemical, and physical properties. Research Paper FPL-RP-680. Madison, WI: U.S. Department of Agriculture, Forest Service, Forest Products Laboratory. 8 p.

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treatment. While results of this study did show penetration of CNC into southern pine and leach-resistant binding of CNC to ADBAC, neither treatment of southern pine with CNC nor the addition of CNC to ADBAC in a dual treatment improved the biological, chemical, or physical durability properties of the wood.

Keywords: Cellulose nanocrystals (CNC), brown-rot fungi, mold fungi, subterranean termites, wood preservation, nanoentrapment, benzalkonium chloride (ADBAC), MOR, MOE, mass loss calorimetry

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Introduction

Leach resistance is a critical factor in development of new wood preservative formulations. Wood preservatives that generally resist leaching during service rely on chemical fixation reactions based on drying time and temperature that take place after the wood is impregnated with the chemical following pressure treatment (Lebow 1996). Wood preservatives that are readily leachable, like borates, have limited applications. Improving the leach resistance of soluble biocides has remained an area of interest to the wood preservation industry prompting the need for novel approaches to wood treatment with leachable preservative chemicals.

Nanomaterials exhibit unique physical and physiochemical properties that differ significantly from larger particles of the same material (Clausen 2007; Clausen et al. 2012). Cellulose nanocrystals (CNC) are reported to demonstrate very high strength and stiffness properties that can impart improved strength to composites and coatings (Wegner et al. 2005; Rains et al. 2014). High-yield cellulose nanocrystal production has recently been achieved on a scalable level through optimization of acid hydrolysis (Wang et al. 2012). Two things happen during acid hydrolysis: First, the amorphous region of cellulose is largely hydrolyzed, but the crystalline regions remain intact, and second, the conditions are such that some of the primary alcohols at the CNC surface are converted to negatively charged sulfate esters. The resulting CNCs are uniformly dispersible; the dispersion stability of CNCs coupled with the controlled uniformity results in a nanomaterial that should be able to penetrate wood in an aqueous dispersion. The negatively charged sulfate ester groups are suitable for ionic binding with cationic quaternary ammonium compounds.

Quaternary and tertiary amine salts, collectively known as alkylammonium compounds (AAC), have long been recognized as effective biocides against most decay fungi and *Reticulitermes* sp. (Preston and Nicholas 1982; Preston et al. 1987) with didecyldimethyl ammonium chloride (DDAC) most commonly used in wood preservation formulations

during the 1990s. In the early 2000s, benzalkonium chloride (ADBAC) was added as a co-biocide to copper monoethanolamine to create one of several alkaline copper quat (ACQ) wood preservative formulations (Lebow 2004). We hypothesize that soluble quaternary ammonium compounds bound to CNC within wood may serve to simultaneously prevent leaching and provide improved physical properties to the wood.

This study had four objectives: 1) screen and select candidate quaternary compounds for optimal binding and efficacy, 2) evaluate efficacy of a CNC/quat hybrid against biological organisms that attack wood in-service, 3) assess penetration of wood and resistance to leaching of CNC/quat hybrid, and 4) determine whether a CNC/quat hybrid alters physical properties of wood following impregnation with the CNC/biocide hybrid molecule.

Materials and Methods

Chemicals

A range of concentrations of four cationic surfactants were screened for antifungal activity against mold and decay test fungi: benzalkonium chloride (ADBAC), cetrymonium bromide (CTAB), and octadecyltrimethylammonium bromide (OTAB) from Sigma-Aldrich, Inc., St. Louis, Missouri, and benzethonium chloride (BZT), Lonza, Inc., Allendale, New Jersey.

Fungal Inoculum

Mold fungi

Three mold fungi, *Aspergillus niger* Tiegh. (2.242) provided by University of Virginia, *Penicillium chrysogenum* Thom. (PH02) from the Center for Forest Mycology Research, Wisconsin, and *Trichoderma atraviride* Bissette (ATCC 20476), were grown on 2% malt extract agar (MEA) (Difco, Becton, Dickinson & Company, Maryland) for 2 weeks. Spore suspensions of test fungi were prepared by washing the surface of each malt agar plate with 10–15 mL of sterile deionized water (DI) according to ASTM standard

Table 1—Binding, stability, and characterization metrics for CNC/biocide hybrid

Biocide concentration (%)	CNC/biocide ratio	Temperature stability (°C)	Binding stability (pH)	Visual observations	Characterization metrics
0.1	1:1	0	3.0	Settling	Dehydration/rehydration
0.5	1:2	10	7.0	Resuspension	EDAX ^a
1.0	2:1	25	9.0	Optics	ICP ^b
2.0		50			SEM ^c

^aEnergy Dispersive X-ray Analysis.^bInductively coupled plasma.^cScanning electron microscopy.

D4445-10 (ASTM 2011). Individual mold spore suspensions were transferred to a spray bottle and diluted to 100 mL with DI water to yield approximately 3×10^7 spores/mL. The spray bottles were adjusted to deliver 1-mL inoculum per spray.

Decay fungi

Brown-rot fungi, *Gloeophyllum trabeum* (Pers.: Fr.) Murr (MAD 617) and *Postia placenta* (Fr.) M. Lars et Lomb (MAD 698), or *Fibroporia radiculosa* (Peck) Gilb. & Ryvarden (TFFH 294) and a white-rot fungus, *Trametes versicolor* (L. Fr.) Pil. (MAD 697) were used to screen chemical and for resistance testing. Test fungi obtained from Forest Products Laboratory, Wisconsin, were maintained on 2% MEA. Agar plugs were taken from the edge of active mycelial growth from individual fungal cultures and separately blended with 10 mL sterile DI water to create a liquid fungal inoculum.

Agar Toxicity Test

One mL of fungal inoculum was sprayed (for mold fungi) or spread (for decay fungi) over the surface of three MEA Petri dishes. Aqueous dilutions of 0.5, 1.0, 2.0, 5.0, and 10% (w/vol) active ingredient were prepared for each cationic surfactant. Filter paper discs (13 mm diameter) were saturated with a 10- μ l aliquot for individual concentrations of test chemicals and placed in the center of the pre-inoculated Petri dishes. Petri dishes were incubated at 27 °C and 70% relative humidity (RH) for 2 weeks or until the control plates were substantially covered with fungal growth. Clearing zones surrounding the chemical-impregnated discs were measured as the zone of inhibition (ZOI) and recorded after subtracting the diameter of the filter paper disc. Based on the results of the agar toxicity test, the most efficacious surfactant (referred to as ADBAC from here forward) was selected for the subsequent experiments.

CNC/Biocide Hybrid Characterization Metrics

Cellulose nanocrystals (CNC) were produced at the Forest Products Laboratory, Wisconsin (Wang et al. 2012). The optimal ratio of biocide to CNC was determined through observation of agglomeration and weight of the CNC/ADBAC

hybrid (Table 1). Visual observations included the amount of settling that occurred upon mixing various ratios of CNC and the biocide, ease of resuspending the settled hybrid and clarity or cloudiness of the sample. To test that biocide efficacy was retained following binding to CNC, equal volumes of CNC were gently mixed with various concentrations of ADBAC and held at room temperature for 24 h. Ten microliters of the resulting CNC/ADBAC hybrid were placed at the center of agar plates that were freshly inoculated with fungal inoculum. Plates were incubated at 27 °C, 70% RH for 3 weeks. ZOI diameters surrounding the CNC/biocide hybrid were recorded when present. Wood treated with the CNC/biocide hybrid was evaluated by energy dispersive x-ray analysis (EDAX), inductively coupled plasma (ICP) and scanning electron microscopy (SEM) for the presence of biocide and CNC.

Temperature stability

Aliquots of the CNC/ADBAC hybrid mixture were placed in four separate tubes and stored at 0, 10, 25, and 50 °C for 24 h. Ten microliters of each aliquot were evaluated for agar toxicity.

pH stability

Aliquots of the CNC/ADBAC hybrid were adjusted with 1N HCl and 1 M NaOH to pH 3.0, 7.0, and 9.0. Ten microliters of each pH-adjusted aliquot were evaluated for agar toxicity as described above.

Wood Treatment

Vacuum impregnation

Southern pine sapwood, 7 × 20 mm cross section × 7 cm long for mold tests, 1 × 1 × 1 cm for decay tests, 25 × 25 × 5 mm for termite tests and 100 mm × 100 mm for mass loss calorimetry were vacuum impregnated in a series of sequential two-step treatments described in Table 2. Each treatment consisted of exposing specimens to the first chemical under vacuum for 20 min at 172 kPa followed by exposing the same specimens to the second chemical under vacuum for an additional 20 min at 172 kPa. Specimens were blotted dry on paper towels between treatments. Controls were subjected to dual treatments with DI water. Following dual

Table 2—Chemical retention in southern pine double treated with CNC or ADBAC

Treatment 1	Treatment 2	Average retention (kg/m ³)
DI H ₂ O	DI H ₂ O	0
CNC	CNC	5.52
ADBAC	ADBAC	18.20
CNC	ADBAC	15.71
ADBAC	CNC	1.98
Pre-mixed CNC/ADBAC	Pre-mixed CNC/ADBAC	2.57

treatment, specimens were air-dried for 72 h prior to conditioning at 27 °C, 70% RH for 2 weeks.

Vacuum-pressure impregnation

Southern pine sapwood stakes (1.9 × 1.9 × 40.6 cm) were submerged in treatment solution 1 (Table 2) for 40 min vacuum followed by 345 kPa for 40 min. Each stake was blotted dry, weighed and submerged in treatment solution 2 (Table 2) for 40 min vacuum followed by 345 kPa for 40 min. Controls were subjected to dual water treatments under the same vacuum and pressure regime. Following dual treatment, specimens were air-dried for 2 weeks and conditioned at 21 °C and 70% RH for 4 weeks prior to testing.

Biological Evaluation of CNC/ADBAC Hybrid Treatment

Mold resistance

Six randomly selected southern pine specimens per treatment group were sprayed with 1-mL spore inoculum and incubated at 27 °C and 80% RH for 4 weeks. Untreated southern pine served as the control. Following incubation, specimens were visually rated on a scale of 0–5 with 0 indicating the specimen was completely free of mold growth and 5 indicating the specimen was completely covered with mold growth.

Decay resistance

Following the guidelines of AWPA standard E10–12 (AWPA 2014), five treated and five untreated southern pine specimens per treatment group were weighed and placed in a soil-block test. *Trametes versicolor* was replaced with *Fibroporia radiculosa* (Peck) Gilb. & Ryvarden (TFFH 294) to test decay resistance of the CNC/ADBAC hybrid in wood blocks due to the unique preservative tolerance properties of *F. radiculosa* of importance to the wood protection industry (Clausen and Jenkins 2011). Soil bottles were prepared by inoculating a pine feeder with one of three brown-rot test fungi, *G. trabeum*, *P. placenta*, and *F. radiculosa*, and incubated at 27 °C and 80% RH for 12 weeks. Following incubation, fungal mycelium was brushed from the specimens,

specimens were oven-dried, reconditioned at 27 °C and 80% RH, and reweighed. Percentage mass loss was calculated for each specimen and reported as an average for each treatment group.

Termite resistance

A no-choice termite test with *Reticulitermes flavipes* (Kollar) (Eastern subterranean termites) was performed using five unleached specimens per treatment and five untreated controls that were pre-weighed and conditioned at 27 °C and 70% RH. Termites were collected from Janesville, Wisconsin. One specimen was placed in the bottom of an acrylic cylindrical container (90 mm diameter and 60 mm tall) with 1 g of *R. flavipes* and moist sand. The containers were maintained at 27 °C and 85% RH for 4 weeks based on AWPA E1–13 standard method (AWPA 2014). Tests were periodically checked for moisture and mortality. At the end of the incubation, wood specimens were oven-dried, reconditioned at 27 °C and 70% RH, and reweighed to calculate mass loss. The termite mortality rate was estimated by counting the remaining live termites. Following incubation, wood specimens were visually rated on a scale of 0–10 with 10 being sound and 0 equaling failure.

Chemical Retention

Analytical assessment

Southern pine specimens that were treated with CNC, ADBAC, or combinations of CNC and ADBAC were assessed with scanning electron microscopy (SEM), EDAX analysis, and inductively coupled plasma (ICP) for treatment penetration and retention following leaching.

Leaching resistance

Leaching procedures were similar to AWPA E11–12 standard method (AWPA 2014). Five specimens per treatment were placed into individual 500-mL beakers, submerged in 100 mL of DI water and subjected to mild agitation for a total of 14 days, with a complete change of water after 6 h, and 1, 2, 4, 6, 8, 10, 12, and 14 days. Leached specimens were dried, reconditioned for 2 weeks at 27 °C and 80% RH, and evaluated for mold and decay resistance. Leached specimens were also subjected to ICP analysis.

Physical Properties

Mechanical properties

Modulus of elasticity (MOE) and modulus of rupture (MOR) were assessed, and work to maximum load (WML) was calculated on 30 specimens for each treatment group according to ASTM D143–14 (ASTM 2014). Work to maximum load is a measure of the combined strength and toughness of the wood under bending stress. Specimens were conditioned at 21 °C and 70% RH for 4 weeks prior to testing. Based on simple fixed effect models, the mean MOE, MOR, and WML for each group of test specimens were compared

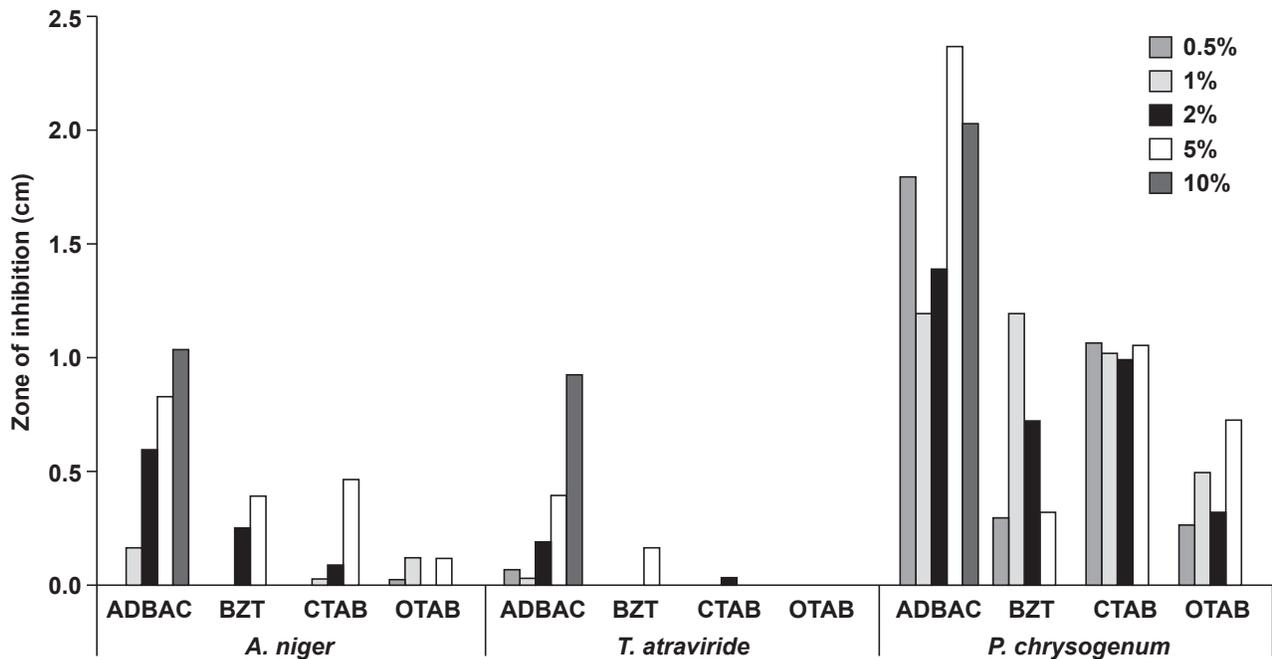


Figure 1—Inhibition of three mold fungi by benzalkonium chloride (ADBAC), benzethonium chloride (BZT), cetrimonium bromide (CTAB), and octadecyltrimethylammonium bromide (OTAB) in an agar toxicity test.

with the mean values of control specimens to determine statistical significance of differences following treatment of wood with CNC, ADBAC, or combinations of CNC and ADBAC using SAS® 9.4.

Combustion characteristics

Specimens for each treatment group were tested in triplicate using the mass loss calorimeter (MLC). The MLC is commonly used as the first step in obtaining combustion and flammability characteristics of materials such as the heat release rates (HRR), effective heat of combustion, and mass loss rates. The MLC uses a truncated conical electric heater that provides a constant heat flux onto a test specimen, and after piloted ignition by the spark igniter, the mass loss and HRR are recorded. The HRR is determined by digitally deconvolving the signal of the chimney wall thermopile and combining the resulting processed signal with the standard gas convection thermopile signal into a composite value (Dietenberger and Boardman 2013). The 100- × 100-mm test specimens were subjected to irradiance at 50 kW/m² in a horizontal orientation. Prior to testing, the samples were conditioned at 21 °C and 50% RH for 4 weeks. The data were analyzed and the averages of the triplicates were compared with the average values of the water-treated control specimens.

Results and Discussion

Agar Toxicity Screening

Agar toxicity test results are summarized in Figures 1 and 2. Of the four cationic quaternary ammonium compounds screened for anti-fungal properties, ADBAC at a

concentration > 2% had the greatest ZOI for the mold fungi (Fig. 1) selected for this study; ADBAC showed the greatest ZOI at all concentrations for the decay fungi (Fig. 2).

Characterization Metrics

Binding characteristics

Visual examination of flocculation patterns for CNC and ADBAC at ratios of 2:1, 1:1, 1:5, and 1:10 after 24 h at 25 °C showed that a 1:1 ratio produced a semi-solid pellet without any unbound liquid (Table 1). At higher ratios of CNC, less than optimal binding occurred resulting in a liquid dispersion with a high flow rate. Lower ratios of CNC to ADBAC resulted in a solid pellet with notable amounts of unbound liquid. The 1:1 ratio was selected for further experiments. Theoretically, CNC/ADBAC agglomeration should facilitate physical entrapment of the otherwise soluble biocide within the wood but without modifying the wood matrix.

Dispersibility is generally essential for CNC storage and transport. Beck et al. (2012) reported that above a threshold moisture content of 4%, dried CNC is fully dispersible in water. The CNC/ADBAC hybrid readily rehydrated after dehydration. Dried preparations of CNC/ADBAC were dispersible, but not necessarily dissociated in water.

Stability tests

For stability tests, CNC/ADBAC was adjusted to pH 3.3, 7.0, or 9.0 before testing for ZOI in a mold inhibition test. Results demonstrated similar ZOIs for mold test fungi at each pH; therefore, pH 7.0 was selected for further experiments. The initial pH of CNC was 7.38.

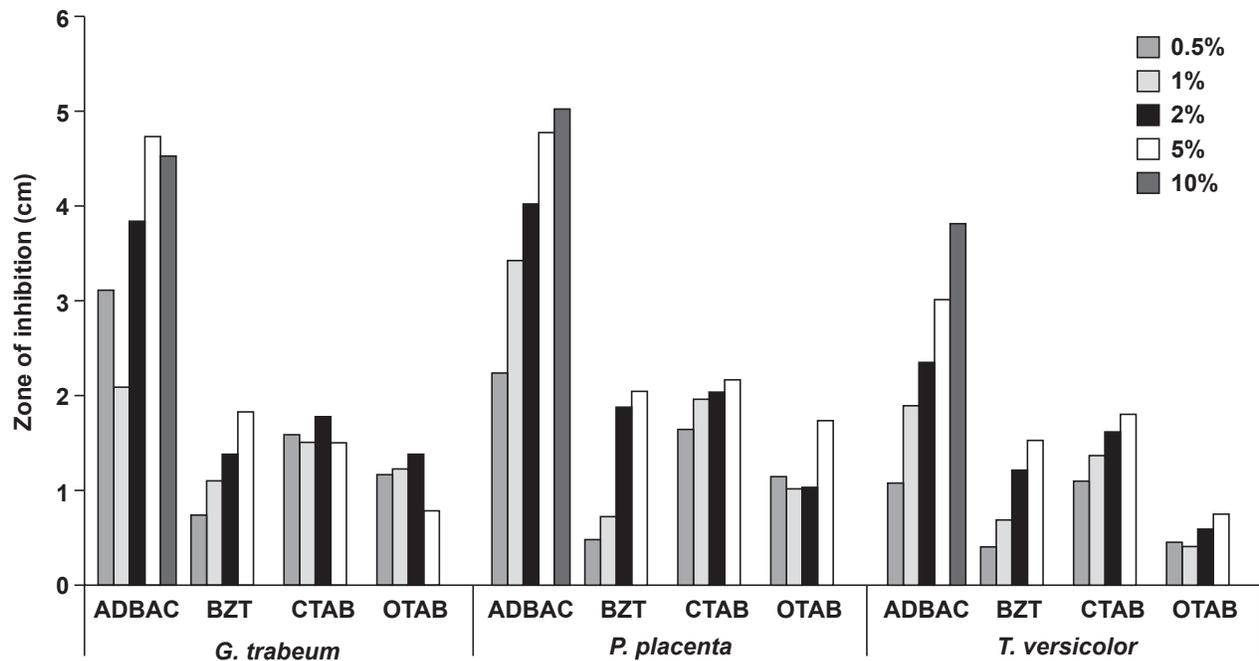


Figure 2—Inhibition of two brown-rot fungi (*G. trabeum* and *P. placenta*) and one white-rot fungus (*T. versicolor*) by benzalkonium chloride (ADBAC), benzethonium chloride (BZT), cetrimonium bromide (CTAB) and octadecyltrimethylammonium bromide (OTAB) in an agar toxicity test.

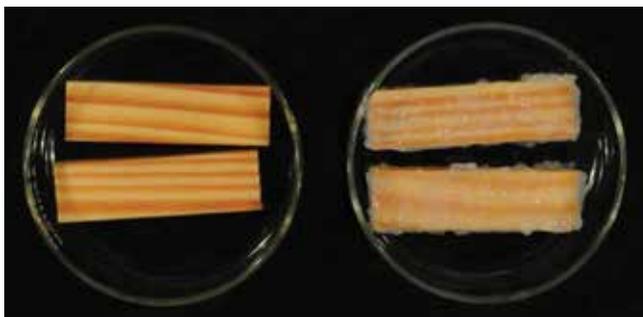


Figure 3—Comparison of penetration for CNC/ADBAC (left) and ADBAC/CNC (right) in southern pine.

CNC/ADBAC was also exposed to temperatures ranging from 0 °C to 50 °C for 24 h before testing for ZOI in a mold inhibition test. Results showed that ZOIs were the same or slightly higher at 25 °C; therefore, 25 °C was selected for further experiments.

Treatment Retention

The dual treatment process consisted of two subsequent treatments in separate chemical solutions (Table 2). The percentage of chemical uptake was calculated. CNC penetrated southern pine resulting in a 5.52 kg/m³ uptake ($n = 5$). Dual treatment with ADBAC and dual treatment with CNC followed by ADBAC resulted in 18.2 and 15.7 kg/m³ uptake, respectively. When wood was treated with ADBAC followed by CNC or when the ADBAC and CNC were pre-mixed before pressure impregnation, the treatment visibly agglomerated on the surface of the wood specimens and prevented appreciable penetration (Fig. 3).

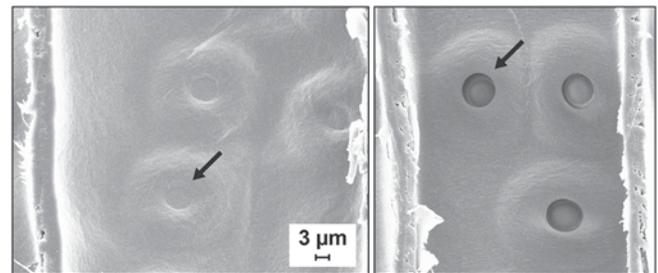


Figure 4—Scanning electron micrograph of southern pine bordered pits (arrow) impregnated with CNC/ADBAC hybrid.

EDAX analysis of treated wood specimens was not sensitive enough to estimate treatment chemical uptake utilizing chlorine as the marker for ADBAC and sulphur as markers for CNC.

Leach Resistance

Binding visibly occurred upon physical contact of CNC with ADBAC to form an agglomeration that appeared to coat the bordered pits of southern pine (Fig. 4). Leached and unleached southern pine specimens treated with CNC and ADBAC were ground to 20 mesh (nominal 841 μm) and analyzed using inductively coupled plasma (ICP) spectroscopy for chlorine and sulphur. The liquid solution of CNC and ADBAC used for treatment was also analyzed. Results showed that approximately 9% of CNC in solution penetrated into wood with 66.7% of the CNC in wood remained after leaching (Table 3). For the ADBAC, 12.6% of the treating solution penetrated the wood with 48% remaining after leaching. Chlorine levels were not detectable in the

Table 3—ICP chlorine and sulphur analysis of unleached and leached southern pine treated with CNC and ADBAC alone and in combination

	Unleached (ppm)		Leached (ppm)	
	Cl	S	Cl	S
Treating solution				
CNC	ND ^a	3,300	–	–
ADBAC	60,900	ND	–	–
CNC/ADBAC	28,700	1,650	–	–
Treated pine				
CNC	ND	300	ND	200
ADBAC	7,700	50	3,700	30
CNC/ADBAC	ND	300	ND	300
Untreated control	ND	40	–	–

^aND, none detected.

CNC/ADBAC mixture in the wood, but 18% of the CNC measured as sulphur was detected in both unleached and leached samples, suggesting the CNC bound to ADBAC within the wood and did not leach out.

Although ADBAC is water soluble, Lee and Cooper (2010) reported two adsorption mechanisms for ADBAC onto wood: ion exchange at low concentration and hydrophobic interaction at high concentrations. They reported that when used without copper, the adsorption capacity of ADBAC through ion exchange does not achieve the Na⁺ cation exchange capacity of wood. The leaching results in this study suggest penetration of the wood by CNC and binding within the wood of CNC/ADBAC. The ADBAC served as the counter cation for the sulfate ester on the CNC and is ionically bound to the CNC, so leach resistance is essentially a measure of the dissociation constant of the CNC-sulfate-ADBAC salt.

Biological Resistance

Treated leached southern pine specimens were tested for resistance to biological attack by mold fungi (Fig. 5), decay fungi (Fig. 6), and subterranean termites (Fig. 7). Results of the mold and decay tests revealed no change in the biological resistance of southern pine when CNC was present in the chemical treatment compared to the chemical treatment alone. All treatments that included ADBAC showed similar levels of fungal inhibition regardless of the treatment group. Results for the termite no-choice test showed a 20% weight loss in water controls, 25% weight loss in CNC controls, and only 2% to 4% weight loss in all samples treated with ADBAC regardless of the treatment group. There was little mortality in the termite test and all specimens that were treated with ADBAC showed visible repellency to termite tunneling.

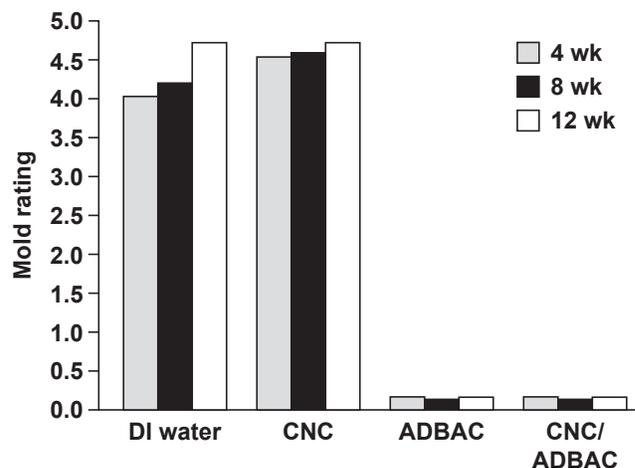


Figure 5—Mold growth after 4-, 8-, and 12-week exposure of leached CNC/ADBAC-treated southern pine to a mixed mold spore suspension.

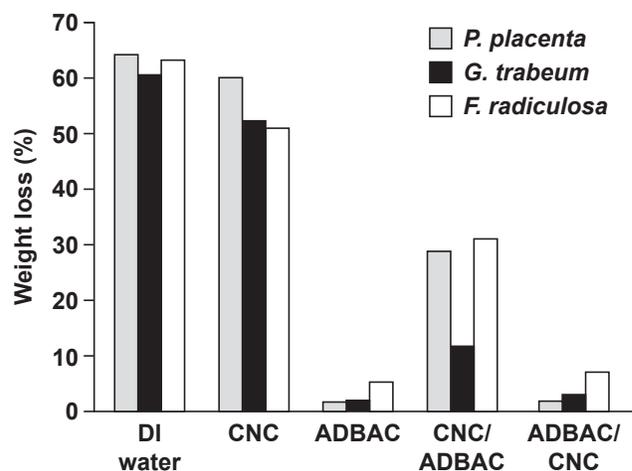


Figure 6—Weight loss in leached CNC/ADBAC-treated southern pine following exposure to *Postia placenta*, *Gloeophyllum trabeum*, and *Fibroporia radiculosa*.

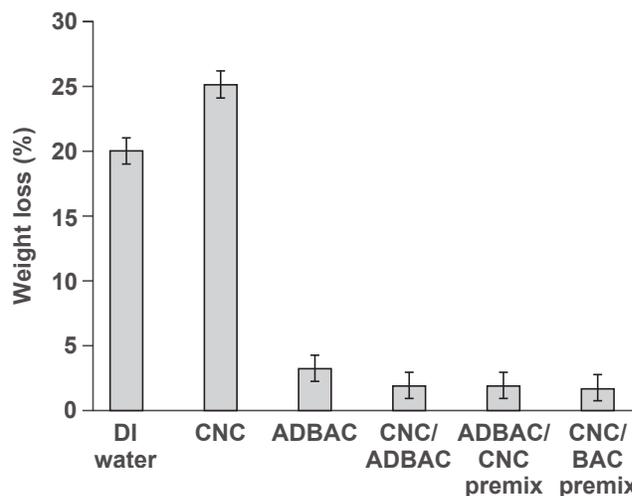


Figure 7—Weight loss in leached CNC/ADBAC-treated southern pine in a no-choice test against *Reticulitermes flavipes* subterranean termites.

Table 4—Mechanical properties of unleached southern pine pressure treated with CNC or ADBAC

Treatment 1	Treatment 2	MOE (MPa)		MOR (kPa)		WML (J)	
		Mean	S.E. ^a	Mean	S.E.	Mean	S.E.
DI H ₂ O	DI H ₂ O	16,500	363.8	112,054	2,298	12.78	0.553
CNC	CNC	16,174	363.8	118,156	2,298	12.54	0.553
ADBAC	ADBAC	17,180	370.0	114,474	2,337	13.06	0.563
CNC	ADBAC	16,450	363.8	114,577	2,298	14.12	0.553
ADBAC	CNC	15,907	363.8	119,079	2,298	12.01	0.553
Pre-mixed CNC/ADBAC	Pre-mixed CNC/ADBAC	16,684	363.8	118,783	2,298	12.64	0.553

^aS.E., Standard Error; note one observation from the ADBAC/ADBAC group experienced a brash tension failure and was excluded.

Physical Properties

Mechanical testing

Mean MOR, MOE, and WML and their associated group estimated standard errors are summarized in Table 4. Group differences were not detected for MOE (p -value = 0.2201), MOR (p -value = 0.1727), and WML (p -value = 0.1537). Results did not reveal any statistical differences between the treatment groups suggesting that penetration of southern pine with CNC under the conditions of this study does not significantly increase or decrease strength, stiffness, or toughness. For each property, a one-way analysis of variance was conducted using SAS® 9.4. Dunnett's test was used to adjust p -values for pairwise comparisons of each treatment group with the controls. Results of the comparison showed no statistical differences between the controls and any of the treatment groups ($p < 0.05$). Agglomerations of CNC in a dispersed solution are reported to weaken the mechanical properties of the CNC (Steele et al. 2012), but it is not known whether agglomeration because of ionic bonding within a wood matrix would demonstrate similar mechanical properties.

Combustion properties

Mass loss calorimetry results showed no appreciable difference between controls and treated specimens regarding effective heat of combustion, mass loss rate, and heat release rates for southern pine pressure-treated with CNC and ADBAC alone and in combination (Fig. 8).

Conclusions

Conditions used to produce cellulose nanocrystals (CNC) result in formation of sulfate ester groups that are suitable for ionic binding with soluble cationic quaternary ammonium compounds. CNC were evaluated for their potential to bind and entrap benzalkonium chloride (ADBAC), a quaternary ammonium compound, in southern pine. Penetration and entrapment of CNC and ADBAC in wood were

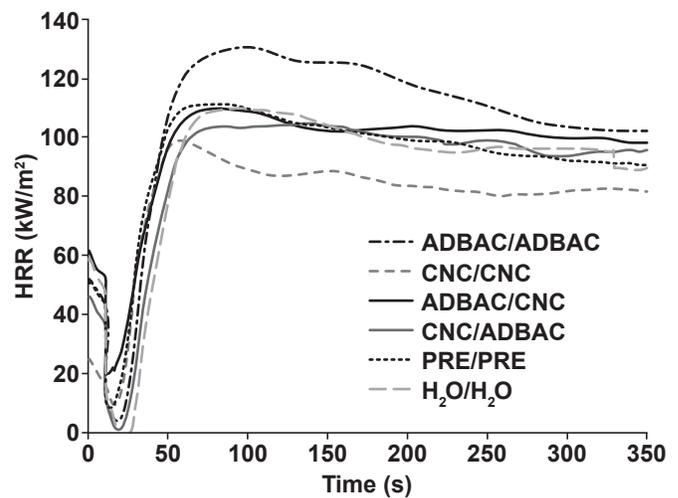


Figure 8—Average heat release rate (HRR) in kW/m² from triplicates of each treatment obtained by the modified mass loss calorimeter.

demonstrated under the conditions of this study. Treated wood specimens were assessed for changes in biological, chemical, and physical properties. It was shown that ADBAC alone provided sufficient efficacy against mold fungi, decay fungi, and subterranean termites in leached specimens. No statistical differences were detected in strength (MOR), stiffness (MOE), or toughness as measured by work to maximum load between control specimens and those that were treated with CNC or ADBAC alone or in combination. Likewise, specimens treated with CNC or ADBAC alone or in combination did not demonstrate changes in combustion as measured by mass loss and heat release rate compared to control specimens. In conclusion, southern pine treated with cellulose nanocrystals agglomerated to benzalkonium chloride (ADBAC) within the wood, but the CNC/ADBAC hybrid molecule does not appreciably alter biological durability, nor does it statistically improve mechanical properties, or combustion resistance compared to untreated controls.

Acknowledgments

Authors thank Steve Halverson, Physical Science Technician, for wood treatments; Tim Nelson, Mechanical Engineering Technician, and Marshal Begel, General Engineer, for conducting mechanical testing and analysis; Kolby Hirth, Chemist, for ICP analysis; and Tom Kuster, Forest Products Technologist, for preparing scanning electron micrographs.

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