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Enhancing the Residual Efficacy of Wood Phytosanitation using a Silane

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ENHANCING THE RESIDUAL EFFICACY OF WOOD PHYTOSANITATION USING A SILANE

By

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PHYTOSANITATION USING A SILANE

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This study investigates use of the organosilane 3-(trimethoxysilyl) propyldimethyl octadecyl ammonium chloride (Si-Quat) as a wood treatment to impart residual moisture and organism control on wood substrates. Study 1, which utilized experimental testing procedures to evaluate mold growth after standardized heat treatment, indicated less surface mold on treated samples. Study 2, which utilized standardized testing procedures to evaluate Si-Quat treated wood's resistance to subterranean termite attack, indicated greater termite mortality and less feeding on treated wood, as well as increased termite feeding preference for untreated wood. Study 3, which utilized standardized testing procedures to evaluate water repellency, indicated significantly reduced moisture gain at higher silane-based treatment levels in comparison to untreated wood. It is concluded that a silane based treatment utilized in this study can be effective for organism control and the possible supplementation to current phytosanitation of wood packaging materials.

DEDICATION

This thesis, representing my pursuit of accomplishment, is dedicated to my grandparents, for without their Christian beliefs, morals, and unconditional love, I would have surely chosen a different path.

Troy Audra Lee Jarnigan ..... (*January 21, 1916 – December 4, 1999*)

Myrtle Marie Jarnigan.....(*February 2, 1922 – February 11, 2010*)

Lance Delano Johnson ..... (*December 25, 1932 – August 22, 1988*)

Tallulah Earline Johnson.....(*February 27, 1933 – Present*)

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CHAPTER I

INTRODUCTION AND LITERATURE REVIEW

Background

Many non-native organisms are transported and dispersed through the human-mediated trade of goods: an estimated 50,000 non-native organisms having been introduced into the United States either intentionally or accidentally (Pimentel *et al*. 2005; Mack *et al*. 2000). Non-native organisms may compete directly with native species for food and nutrient sources, threaten biodiversity, affect native ecology, and may cause substantial economic loss (Mack *et al*. 2000; Mumford 2002; Pimentel *et al*. 2005; Work *et al.* 2005). Many organisms, however, are beneficial to humans and have been successfully integrated into the United States fabric including food crops such as corn, wheat, rice, and livestock species such as cattle and poultry. These beneficial, agricultural non-natives provide over 98% of the U.S. food supply (Pimentel *et al*. 2005). However, the potential threat associated with the spread of non-native species is correlated with the establishment, propagation and favorable habitat of each species. Collectively, non-native species including mammals, birds, amphibians and reptiles, fish, arthropods, mollusks, weeds, vertebrate pests, insects, mites and plant pathogens cause significant damages annually. From 1906 to 1991 there was an estimated $97 billion worth of damages from exotic pests (OTA 1993).

Because raw wood, as well as most wood products, are favored food sources and harborage material for varying types of organisms, wood and wood products are often the medium of accidental transport from one geographic location to another for exotic forest insects (EFI) as well as wood-destroying insects (WDI). International trade is the primary method by which non-native insects are dispersed between countries, and wood packaging materials (WPM) have been the associated source and major contributor of many non-native fungi and bark- and wood-destroying insect introductions into the U.S. (Pasek *et al*. 2000; USDA 2000; McCullough *et al*. 2006; Colunga-Garcia *et al*. 2009)

In a study conducted by McCullough, an estimated 725,000 pest interceptions were recorded between 1984 and 2000 in the Port Information Network (PIN). This database, maintained by the U.S. Animal and Plant Health Inspection Service, Plant Protection and Quarantine division (APHIS, PPQ), documents daily non-native species interceptions along borders and ports of entry (Work *et al*. 2005; McCullough *et al*. 2006). One study presented by R.A. Haack documents 25 new species of bark- and wood-destroying Coleoptera (beetles) that have been intercepted in the continental U.S. between 1985 and 2008 (Haack 2006). Haack reported that most of the Coleoptera (beetles) were intercepted on crating, dunnage and pallets. Other non-native species introductions include the discovery of *Xyleborus maiche* (Stark), an ambrosia beetle collected from funnel traps in Pennsylvania, as well as the Buprestid *Agrilus subrobustus* (Saunders) found in Georgia (NPAG 2006; Westcott 2007). The more common non-native species associated with WPM introduced to the U.S. include the Formosan subterranean termite, *Coptotermes formosanus* (Shiraki), the emerald ash borer, *Agrilus planipennis* (Fairmaire), the Asian longhorned beetle, *Anoplophora glabripennis*

(Motshulsky), the sirex wood wasp, *Sirex noctilio* (Fabricius), as well as many ambrosia and bark beetle species. Non-native species have the potential to cause significant agricultural, ecological and economic losses, and are targeted in quarantine efforts of trade goods as well as shipping medium.

Wood pallets and wood containers consist of wood or wood products utilized to support, protect or carry a commodity (IPPC 2008A). Each year the wood pallet and wood container industry utilizes a substantial amount of lumber from hardwood and softwood trees, which is a primary food source for many insects as well as mold and decay fungus species. In 1995 approximately 6.31 billion board feet (MMBF) of lumber were used to produce 411 million new pallets, with similar utilization of material occurring in 1999 where an estimated 6.54 MMBF were used in pallet production (Reddy *et al*. 1997; Bejune *et al.* 2002; Molina-Murillo *et al*. 2005). Since wood packaging materials are renewable as a forest product, are low cost to produce, and easy to handle, they are valuable commodities in today's shipping industry and may not be easily replaced. However, the current concern with the unintentional transport of non-native species has quite literally put wood under the microscope as a carrier of many non-native species during the transport of goods both domestically and internationally, making the quarantine and phytosanitization of WPM a necessity.

International Standards for Phytosanitary Measures

Phytosanitization Measure was the term given by the Food and Agricultural Organization (FAO) and is defined as:

> *"Any legislation, regulation or official procedure having the purpose to prevent the introduction and/or spread of quarantine pests, or to limit the economic impact of regulated non-quarantine pests."* (IPPC 2008A)

In March of 2002 an international standard for phytosanitary measures (ISPM) was developed and titled "Guidelines for Regulating Wood Packaging Material in International Trade", ISPM 15, by the Interim Commission on Phytosanitary Measures (IPPC 2002; Molina-Murillo 2005). The Interim Commission of Phytosanitary Measures is the regulating authority for the International Plant Protection Convention (IPPC). These international standards outline the approved measures by which WPM may be sanitized to prevent the spread of quarantined pests, as well as to outline regulatory and operational requirements and product marking for approved treatment. Measures approved in ISPM 15 for the phytosanitation of WPM include heat treatment (HT) as well as fumigation with methyl bromide (IPPC 2009).

Heat Treatment

Historically, "heat treatment" refers to the utilization of intense, sustained heat to alter wood properties, stabilize its dimensions, and decrease its attraction to water and water permeability, thus increasing resistance to fungal decay (Stamm 1946, 1960; Seborg *et al*. 1953; Ahola *et al.* 2002; Vukas *et al*. 2010). As a result of this process, it has been reported that a decrease in dimensional change (shrink/swell) and equilibrium

moisture content (EMC) of as much as 50% can be achieved when wood is heated to temperatures of 160-260 °C (320-500 °F) (Vukas *et al*. 2010). However, the use of heat, at considerably lower temperatures, has also been studied intensively for many years as a means of controlling insects. Most insects have a comfortable operating range between 0-45 °C (32-113 °F), and surpass their thermal limits if cooled below or heated above this range for any length of time (Fields 1992; Wright *et al*. 2002). For example, studies conducted as early as 1883 revealed that temperatures ranging from 48.8-60 °C (120-140 °F) were fatal to the larva of the Angoumois grain moth, with certain exposure times (Dean 1911). Another study in 1924 by Snyder and St. George concluded that, in regard to the powder-post beetle *Lyctus planicollis* (LeConte) in ash and oak lumber, wood core (centers of HT pieces) temperatures above 54 °C (130 °F) maintained for one and one half hours are fatal, but temperatures below 54 °C are not (Snyder and St. George 1924). Extensive time/temperature schedules to heat certain media (*e.g*. grain and wood) to eradicate a wide variety of insects have been compiled (Strang 1992). Studies have not only been focused on the time/temperature schedules of insects infesting media, but also in the control of insects infesting entire structures (Forbes 1987, 1989). The ability to thermally eradicate organisms infesting certain media (*e.g*. the time to which the centers of pieces are heated to specific temperatures) defines the "heat treatment" that will be discussed in this manuscript.

Heat Treatment in ISPM 15

Heat treatment specifications outlined in ISPM 15 require that WPM be heated to insure a minimum core temperature of 56 °C (132.7 °F) for a minimum time of 30 minutes, a time/temperature schedule referred to in the industry as 56/30. This specific time-temperature schedule was chosen with consideration of published research and data on insect mortality for a wide variety of pests as well as it's commercially feasibility. This time-temperature schedule is not sufficient for all pests however. For example, ash infested by the emerald ash borer requires a heat treatment where the core temperature reaches 60 °C (140 °F) and is held for 60 minutes (USDA 2011).

Kiln-drying (KD), heat enabled chemical pressure impregnation (CPI), and microwave are accepted as heat treatments for WPM, given that the specific time/temperature schedules used meet the minimum HT requirements of 56/30 as outlined in ISPM 15 (IPPC 2009). ISPM 15 standards require that all WPM undergoing a form of treatment be clearly marked and identified as receiving such treatment, so that inspectors can insure that such treatments have been made. (Figures **1.1** and **1.2)** Minimum marking requirements are to include the IPPC symbol, country code (two letters), producer number assigned by the NPPO, and the IPPC abbreviation for treatment received (e.g., HT or MB). Regarding standardization, the American Lumber Standard Committee (ALSC) issues grade stamps for the two heat treatment categories: Kiln-dried heat treated (KD HT) and non-kiln dried heat treated (HT) (Wang 2010).

The KD process is usually completed when the wood moisture content (MC) is reduced from green (100%) to below 19% (dry-basis). Provided the wood MC does not increase after KD, the removal of moisture during a KD cycle typically prevents re-

infestation of wood by insects and other organisms which require certain moisture levels for survival as well as optimal life cycle completion. For HT wood, on the other hand, the moisture level in the wood may not be sufficiently decreased during the treatment process to the lower 19% MC, leaving the wood susceptible to re-infestation by insects, decay and surface mold fungi. During HT, the wood surface and core temperature rises, with moisture migrating from the core to the surface. Combined with the elevated temperatures during the cooling process, the wood can be susceptible to mold (Denig and Bond 2003). This susceptibility may be prevented with the use of a complete KD cycle which dries the wood to below 19%; however, with the volume of WPM requiring ISPM 15 treatment before shipment, the KD process is impractical due to the associated energy costs and time required, further illustrating the need for an energy-efficient treatment method providing long-term control over re-infestation by organisms.

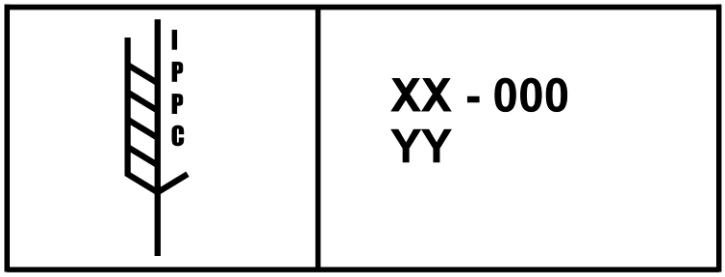

Figure 1.1

Certification mark on wood packaging material that has been subjected to an approved treatment outlined in ISPM 15.

Figure 1.2

Certification mark showing the IPPC symbol, two-letter country code, NPPO assigned producer number, and treatment abbreviation for HT.

Fumigation

Along with HT, fumigation with methyl bromide (MeBr) is a currently approved measure in ISPM 15 for the phytosanitization of WPM (IPPC 2009). The efficacy of MeBr to eradicate insects infesting various forms of media by fumigation is widely known (Bess and Ota 1960; Hanula and Berisford 1982; Yang *et al*. 1995; Donahaye 2000; Barak *et al*. 2005). Methyl bromide, also known as bromomethane and monobromomethane, has been used as an industrial chemical in the U.S. since the 1920's and was commonly used as a fire extinguishing agent in submarines and airplanes in World War II (Prain and Smith 1952). Later, MeBr evolved in use as a soil fumigant and as an insecticidal fumigant for structures and transportation material (CAS 74-83-9; Yang *et al*. 1995). In 1999 over 75% of the MeBr was used for soil fumigation (Ristaino and Thomas 1997; UNEP 1998). In addition to anthropogenic uses, MeBr is also emitted naturally, primarily from oceans and plants (Anbar *et al*. 1996; Gan *et al*. 1998). Methyl bromide is regulated in ISMP 15 based on a minimum temperature/dosage/time schedule to achieve exposures to certain concentration levels with specified monitoring intervals. (Table **1.1**)

Table 1.1

Example of a treatment schedule for MeBr fumigation of wood packaging material to meet the minimum requirements as specified in ISPM 15.

Temperature	Dosage (g/m^3)	Minimum concentration (g/m^3) at:		
		2 h	4 h	24 h
21 °C or above	48	36	31	24
16 °C or above	56	42	36	28
10 °C or above	64	48	42	32

Though effective for the eradication of many organisms, MeBr poses serious risks to both the environment and to human health. In 1991, MeBr was identified as a Class 1 ozone depleter with an ozone depletion potential of 0.65 (Albritton and Watson 1992). This 0.65 ranking indicates that MeBr has 65% of the ozone depleting potential as trichlorofluoromethane (CFC-11). Methyl bromide undergoes photo-oxidation in the stratosphere which releases the bromine atoms; from there the bromine reacts with ozone which results in depletion of the stratospheric ozone layer (Pyle *et al*. 1991). Regulations were set in place as of January 1, 1996 to phase out the use of MeBr, and since 2005 it has been phased out entirely in many developed countries, except as a sanitizing fumigant for international shipping (UNEP 2000). Human exposure to MeBr by either inhalation or through the epidermis or mucous membranes can cause a host of health effects including headaches, nausea, vomiting, confusion, behavioral disturbances, convulsions, and coma (van den Oever *et al*. 1984; Deschamps and Turpin 1996). A possible neurological effect of MeBr exposure is through the inhibition of enzymes and proteins because MeBr is a methylating agent (Torkelson and Rowe 1981).

Because of the numerous health and environmental concerns associated with MeBr, the ISPM recommended in 2008 that the usage of MeBr should be replaced or reduced (IPPC 2008B). Additionally, the European Union banned MeBr as a phytosanitation procedure in 2008 (Vassiliou 2008). Replacements for MeBr are being sought in the form of other fumigants (Yang *et al*. 1995; Barak *et al.* 2006). These factors, along with the fact that MeBr does not offer any residual protection to the medium treated (fumigated), present an opportunity to develop effective, long lasting, environmentally safe alternatives to replace the use of this toxic substance (Donahaye 2000).

Alternative Treatment Methods

Many alternatives for the eradication of organisms infesting raw wood and wood products have been evaluated. The use of vacuum treatments have been studied on insects since the invention of the mechanical air pump by Robert Boyle and continue to be evaluated today as an environmentally benign method of sanitizing wood and wood products (De Bellesme 1880; Back and Cotton 1925; Chen *et al*. 2006). The general consensus for many sanitization methods is that elevated temperatures for a duration of time is sufficient to eradicate most organisms, hence the acceptance of HT into ISPM 15. Though the use of kilns or ovens is the commonly accepted method to raise wood temperatures, other methods have been evaluated to raise internal temperatures of solid wood to meet ISPM 15 by using liquids, including using the biocide disodium octaborate tetrahydrate (DOT) at elevated temperatures to reach the minimum time/temperature

requirements in ISPM 15, as well as provide residual protection with the DOT (Slahor *et al.* 2005; Taylor and Lloyd 2009).

Mold

Like other organic materials, wood is susceptible to colonization by fungi given favorable growth conditions (Davidson 1935; Dowding 1970; Robbins and Morrell 2002). Some media only serve as a host for particular types of fungi, while other media are susceptible to colonization by a wide variety of fungi (Bowyer *et al.* 2003). Wood inhabiting fungi can be categorized by the type of damage they incur: decay, stain and mold. Decay fungi degrade the cell walls of wood, thus causing loss of structural integrity, and have the potential to completely destroy colonized wood. Staining fungi cause discoloration of sap-wood and significantly reduce aesthetic appeal, but do not cause significant strength damages. Mold fungi colonize the sap-wood and produce pigmented spores on the surface, thus also affecting aesthetic appeal, but with negligible effect on wood properties. (Forest Products Laboratory 1999; Bowyer *et al*. 2003)

Temperatures favorable for plant growth are favorable to decay fungi as well, and severe decay occurs when wood is above its fiber saturation point (FSP) (cell walls are full of water), 30% MC (Forest Products Laboratory 1999). Conversely, decay fungi and molds can be prevented by the removal of water from wood when it is dried to below 20% MC and kept dry (Forest Products Laboratory 1999). Traditionally, in an attempt to control fungal growth on WPM manufactured from unseasoned wood, prophylactic fungicides (*e.g*., antisapstain treatments) are applied either by dipping or spraying directly to wood surfaces (Xiao and Kreber 1999). Mold fungi are a prevalent concern regarding

wood and wood products. Consumer perception of mold in general, heightened by news media coverage and attention to the infamous "toxic mold", has created problems in the shipping industry since those receiving shipments are unwilling to accept molded WPM. The improbability that HT at the time/temperature schedule outlined in ISPM 15 will sufficiently reduce the MC of WPM, as well as the likelihood that WPM undergoing a sufficient KD cycle will be placed in conditions that allow the MC to increase afterwards, creates the need for a sustainable treatment that will inhibit mold growth.

Objective

It is specified in the scope of the ISPM 15 standard that the phytosanitary measures sanctioned in the standard are not intended to provide ongoing protection from pests and organisms. Re-treatment of WPM (*e.g.* pallets and crates) is required if the WPM is altered in any way as to suggest that every component of the material has not received an approved treatment. The recent media attention over product recalls involving "moldy" pallets has resulted in increased scrutiny of treatment methods which have brought into question the transport of molds and other fungus species (Staff 2010). Even though the WPM has undergone ISPM 15 treatment, the moisture level often may be sufficiently high enough for infestation by organisms (*e.g.*, non-native species such as EFI and WDI) to occur once it has cooled (Bond 2005). Reinfestation of WPM by organisms after treatment, in part due to the allowance of bark on WPM by ISPM 15, is common, and it is often visible in the form of fungi, frass, soil, as well as live insects (Haack and Petrice 2009; Zahid *et al*. 2008).

It is the hypothesis of this research that wood treatment with the organosilane 3-(trimethoxysilyl) propyldimethyloctadecyl ammonium chloride, (Si-Quat), can supplement the phytosanitation of wood by providing residual moisture control and toxicity to organisms. Experiments conducted evaluate Si-Quat's efficacy as a moldicide (capable of maintaining efficacy after exposure to HT), water repellent, and termiticide on wood substrates, while reviewing its use in conjunction and comparison with the known biocides, disodium octaborate tetrahydrate (DOT) and didecyl-dimethylammonium chloride (DDAC).

CHAPTER II

LABORATORY TESTS TO EVALUATE THE EFFICACY OF GUM (*LIQUIDAMBAR STYRACIFLUA*) TREATED WITH SI-QUAT AND SUBSEQUENTLY HEAT TREATED AGAINST MOLD FUNGI

Introduction/Literature Review

This study was conducted to evaluate the efficacy of a non-leaching antimicrobial organosilane, 3-(trimethoxysilyl) propyldimethyloctadecyl ammonium chloride, (Si-Quat), to inhibit mold growth on unseasoned gum (*Liquidambar styraciflua*) lumber. Si-Quat, a quaternary ammonium chloride (QAC) and silane quaternary ammonium salt, has been extensively documented for its use on textiles and as a sanitizing agent in sterile environments (*e.g*., hospitals) because of its antimicrobial properties (Hayes and White 1984; Kemper *et al*. 2005; Monticello *et al*. 2009).

Many products currently marketed and labeled for use on wood products to inhibit mold fungi (*e.g*., Boracare with Mold Care, Bardac 2280, NP-1, F2, Ecobrite III, and Timbercoat II) contain the bactericide/fungicide/biocide didecyl-dimethylammonium chloride, DDAC. Utilized extensively in the protection of freshly cut lumber from a host of organisms including mold, decay and sapstain fungi, as well as insects, DDAC is a key ingredient in 95% of the sapstain control products utilized in Canada (Chen *et al.* 1995). Also a QAC or alkylammonium compound (AAC), DDAC is a fungicidal component of the commercial wood preservative ammoniacal copper quat (ACQ) (Chen *et al*. 1995;

Hwang *et al*. 2006). Studies have shown DDAC to successfully reduce surface mold on both pine and aspen wood species (Micales-Glaeser *et al*. 2004)

QAC's

Quaternary ammonium chlorides have been utilized as microbiocides for many years and are known for their ability to protect substrates from many bacteria, fungi, and algae (Butcher *et al*. 1977; Chen *et al*. 1995). For example, data compiled by Butcher shows that a wide variety of QAC's at variable concentrations are effective in preventing decay of *Pinus radiata* (Butcher *et al*. 1977). These compounds (QAC's) are common components in household disinfecting agents and sanitizers, algaecides for swimming pools, as well as fabric softeners and conditioners due to their low mammalian toxicity (Nicholas *et al*. 1991; Hwang *et al*. 2006).

Much data has been published on the use of QAC's (*e.g.*, DDAC) on wood substrates to inhibit growth of fungi; however little data is available on the utilization of the QAC 3 (trimethoxysilyl) propyldimethyloctadecyl ammonium chloride, (Si-Quat) on wood substrates. The two modes of action by which these two QAC's provide microbial protection are different in nature. The ability of DDAC to leach is well documented, and, therefore, its use is best suited in non-ground contact applications (Nicholas *et al*. 1991; Hwang *et al*. 2006). However, Si-Quat is categorized as a non-leaching antimicrobial and is capable of covalently bonding to substrate surfaces (Isquith *et al.* 1972; Walters *et al*. 1973; Speier and Malek 1981; Monticello *et al*. 2009). The concept of a covalently bonded, non-leaching antimicrobial utilized on wood packaging material (WPM) has promise to offer residual protection against microbes on wood substrates. Along with

covalent bonding potential, the presence and degree of treatment with Si-Quat is verifiable by the use of an indicator, bromophenol blue. This qualitative indication can be assessed at the time of the mandatory, regulated product markings for WPM receiving ISPM 15 treatment.

Background

Previous, unreported experiments by the author were conducted in which a test method was evaluated where specimens from a representative hardwood species (gum) and softwood species (pine) were treated with various chemical formulations and placed in non-sterile testing conditions with controlled temperature and humidity to accelerate mold growth. The results of those experiments indicated a viable test method with which to evaluate the efficacy of mildicides on wood substrates exposed to favorable mold growth conditions. The present study will replicate the methods used in the previous experiments and evaluate the ability of Si-Quat to inhibit mold growth on wood substrates when exposed to cyclic heat treatments per ISPM 15 standards.

There are standardized test methodologies for evaluating mold growth on wood substrates, such as the American Wood Preservers Association E24-06 Standard Method of Evaluating the Resistance of Wood Product Surfaces to Mold Growth (AWPA 2010A), and the American Society for Testing and Materials D 4445-03 Standard Test Method for Fungicides for Controlling Sapstain and Mold on Unseasoned Lumber (Laboratory Method) (ASTM 2010), however, the experimental testing protocol utilized in the previous experiment served as the testing protocol in this experiment.

Experimental

Wood materials utilized in this study were cut from a single pine and a single gum tree harvested from the Mississippi State University Dorman Lake test site. The trees were harvested and the bucked log sections were transported back to the Mississippi State University Forest Products Laboratory approximately 24 hours after felling. The log sections were stacked on a concrete pad, end sealed, and placed under a water sprinkler to slow drying until use in testing. Logs remained in water storage for approximately two weeks before use. A portable sawmill was utilized to cut 1 in. (2.5 cm.) boards from logs of both tree species. Test specimens were then cut from the gum boards with final dimensions measuring 7 in. x 0.7 in. x 0.75 in., (17.78 cm. x 1.90 cm. x 1.90 cm.), (L x R x T). Specimens were end-sealed on both ends to provide accurate treatment results and marked with a line 3 in. (7.62 cm.) from either end, longitudinally. One end (3 in., 7.62 cm.) was used as a control end and did not receive any treatment. The opposing end (3 in., 7.62 cm.) received treatment, and the middle portion (1 in., 2.54 cm.) allowed for solution wicking and was not evaluated during testing. (Figure **2.1)** As shown in Table **2.1**, this study consisted of 13 treatment groups with 5 replicates per group.

Figure 2.1

Specimens marked three inches from either end.

Table 2.1

Treatment groups represented in testing, percent active ingredient used, target treatment level, and the actual treatment level for each group.

#	Treatment Group[A]	% ai[B]	Target mg ai/sqft[C]	Actual mg ai/sqft[D]
1	DOT Dip	10	-----	-----
2	DDAC Dip	1.5	-----	-----
3	Boracare® with Moldcare®	-----	-----	-----
4	Low Conc. Si-Quat Spray	2.5	100	112
5	Low Conc. Si-Quat Dip	2.12	100	118
6	Low Conc. Si-Plus Spray	2	100	118
7	Low Conc. Si-Plus Dip	2	100	83
8	High Conc. Si-Quat Spray	2.5	150	177
9	High Conc. Si-Quat Dip	3.18	150	146
10	High Conc. Si-Plus Spray	2	150	157
11	High Conc. Si-Plus Dip	2	150	122
12	DOT + High Conc. Si-Plus Spray	10 / 2	--- / 150	--- / 161
13	DOT + High Conc. Si-Quat Spray	10 / 2.5	--- / 150	--- / 173

[A] DOT = disodium octaborate tetrahydrate, DDAC = didecyl-dimethylammonium chloride. Boracare with Mold Care = provided by Nisus, Si-Quat = 3 trimethoxysilyl propyldimethyl octadecyl ammonium chloride in a 42% ai methanol solution diluted to final treatment solution with deionized water, Si-Plus = Si-Quat with proprietary additive in a 2% ai solution, DOT + = DOT treated test samples over-sprayed with either Si-Plus or Si-Quat.

[B] Percent solutions were calculated on a wt/wt basis.

[C] Si-Quat/Plus manufacturer recommended application level.

[D] Actual treated application level.

Application Methods

All test specimens were non-seasoned prior to treatment. Two methods of chemical application were evaluated during this study to simulate application procedures likely to be utilized in industry (Xiao and Kreber 1999). Some test samples were treated by dipping in solution to simulate the utilization of dip tanks in a WPM manufacturing facility. Other specimens were treated by spraying with solution to simulate the

utilization of pneumatic chemical application during conveyance from one process to the next in a WPM manufacturing facility.

For silane-based and DDAC dip-treatment groups, the non-control end of test samples were momentarily dipped in solution, while the non-control end of test samples for DOT and Boracare® with Mold Care® treatment groups received a 30 second dip . (Figure **2.2**) The momentary dip for silane-based groups was based on an experiment by the author in which gum samples were dipped for 5 different time increments; momentary (0 sec.), fifteen seconds (15 sec.), thirty seconds (30 sec.), forty-five seconds (45 sec.) and sixty seconds (60 sec.). Statistical analysis of three replicates of each time increment showed no significant difference at a 95% confidence level in the average milligrams (mg) of active ingredient (ai) retained from each test sample in a single solution of 1.5% ai Si-Quat among the five (5) dip times.

Treatment groups requiring a spray application were sprayed with a VAPER™ Gravity Feed HVLP Touch-Up Spray Gun (Model 19110). Compressed air at fifty (50) pounds per square inch (psi) maintained pressure in the gun. Trial samples were used to adjust the solution delivery and spray pattern to achieve a uniform coating. For this application, the non-control ends of test samples were treated by spraying the radial and tangential surfaces (R x T) to provide an even coating of solution. Since all test samples were end-sealed prior to treatment, no solution was deliberately sprayed on the longitudinal (L) surface.

Figure 2.2

Test sample receiving a 30 second dip of one end.

Treatment

Control ends of test specimens receiving a dip treatment were submerged in deionized water momentarily and removed while control ends of specimens receiving a spray treatment were sprayed with deionized water so that a uniform wetness was evident. Water uptake for the two treatment methods, dip and spray, was calculated and the mean values were used to determine solution ai for chemical treatment groups. The target treatment levels of silane-based solutions were based on the Si-Quat and Si-Plus manufacturer's recommendation of applying 100 milligrams of active ingredient per square foot (mg. ai/sqft) or 150 mg. ai/sqft. There was no target retention for treatment with DOT, DDAC, or Boracare® with Mold Care® treatment groups.

Treatment groups one (1) and three (3) were dip-treated with DOT and Boracare® with Mold Care®, respectively. (Table **2.1**) DOT was mixed with water at 10% ai and the Boracare® with Mold Care® was a ready to use (RTU) product. With treatment group 1,

the non-control ends of 5 replicates were dipped for 30 sec. in the 10% ai DOT solution. With treatment group 3, the non-control ends of 5 replicates, were dipped for 30 sec. in the Boracare® with Mold Care® solution. For treatment group two (2), the non-control ends of 5 replicates were dipped momentarily in a 1.5% ai DDAC solution.

Treatment groups four (4) and eight (8) were spray-treated with a Si-Quat solution. For all Si-Quat spray treatments, a 2.5% solution was used. The mg. ai/sqft for each treatment group was adjusted based on the sample weight after treatment. (i.e., more mg. ai/sqft were delivered to higher concentration treatment groups by applying more chemical). With group 4, a low concentration Si-Quat spray, the non-control ends of 5 replicates were sprayed with 2.5% ai Si-Quat solution to achieve a target treatment level of 100 mg. ai/sqft (actual group average was 112 mg. ai/ sqft). With group 8, a high concentration Si-Quat spray, the non-control ends of 5 replicates were sprayed with 2.5% ai Si-Quat solution to achieve a target treatment level of 150 mg. ai/ sqft (actual group average was 177 mg. ai/sqft).

Treatment groups five (5) and nine (9) were dip-treated with a Si-Quat solution. Based on the average amount of water uptake by control ends for all gum test specimens, 2.12% ai and 3.18% ai Si-Quat solutions were used to dip-treat gum samples momentarily for group 5 (100 mg. ai/sqft target, 118 mg. ai/sqft actual) and group 9 (150 mg. ai/sqft target, 146 mg. ai/sqft actual).

Treatment groups six (6) and ten (10) were spray-treated with a Si-Plus solution, a proprietary solution containing the active ingredient in Si-Quat. The Si-Plus chemical was received from the manufacturer in the form of a 2% ai solution; therefore a 2% ai solution was used for all Si-Plus spray treatments. The mg. ai/sqft for each treatment

group was adjusted based on the sample weight after treatment. With group 6, a low concentration Si-Plus spray, the non-control ends of 5 replicates were sprayed with 2.0% ai Si-Plus solution to achieve a target treatment level of 100 mg. ai/sqft (actual group average was 118 mg. ai/ sqft). With group 10, a high concentration Si-Plus spray, the non-control ends of 5 replicates were sprayed with 2.0% ai Si-Quat solution to achieve a target treatment level of 150 mg. ai/ sqft (actual group average was 157 mg. ai/sqft).

Treatment groups seven (7) and eleven (11) were dip-treated with a Si-Plus solution. A 2.0% ai Si-Plus solution was used to dip-treat gum test samples for both treatment groups. With group 7, a low concentration Si-Plus dip, the non-control ends of 5 replicates were dip-treated momentarily to achieve a target treatment level of 100 mg. ai/sqft (actual group average was 83 mg. ai/sqft). With group 8, a high concentration Si-Plus dip, the non-control ends of 5 replicates were dip treated to achieve a target treatment level of 150 mg. ai/sqft. Because using a concentration greater than 2.0% ai was not possible, treatments were made on a sample-to-sample basis in which varying dip times were used to achieve higher treated weights (actual group average was 122 mg. ai/sqft).

Treatment groups twelve (12) and thirteen (13) were each dip-treated with DOT followed by a spray treatment with Si-Plus and Si-Quat respectively. With groups 12 and 13, the non-control ends of 5 replicates were dip-treated for 30-sec. in a 10% ai DOT solution. With group 12, the non-control ends of 5 replicates, DOT treated, were spray treated with a 2% ai Si-Plus solution to achieve a target treatment level of 150 mg. ai/sqft (actual group average was 161 mg. ai/sqft). With group 13, the non-control ends of 5

replicates, DOT treated, were spray-treated with a 2.5% ai Si-Quat solution to achieve a target treatment level of 150 mg. ai/sqft (actual group average was 173 mg. ai/sqft).

Once all test samples were treated for testing, they were placed in an oven for 30 minutes at 100 °C as per the curing process recommended by the silane-based chemical manufacturer. The testing procedure for this study is separated into two cycles. Each cycle outlined in the study consists of a heat treatment (HT) of the treated test specimens, subjection to accelerated mold growth conditions, evaluation of surface mold growth and test specimen cleaning.

Cycle 1

Treated test specimens were transferred to a laboratory kiln for heat treatment approximately 24 hours after chemical treatment. Two specimens were randomly selected for the attachment of thermocouples to monitor both surface and core temperatures throughout the HT process. For the specimen receiving a thermocouple for core temperature monitoring, a hole of equal diameter to the diameter of the thermocouple wire was drilled half the depth into the specimen along the midpoint of the sample length. The thermocouple wire was inserted into the drilled hole and held in place by a push-pin. For the specimen used to monitor surface temperature, a thermocouple wire was pinned to the surface of the specimen along the midpoint of the length using a push-pin in such a way so that the exposed end of the thermocouple rested in direct contact with the surface of the test specimen. (Figure **2.3**)

Unseasoned pine boards were stacked, un-stickered, in the kiln to fill void space in the kiln and aid in achieving desired wet bulb temperatures. Test specimens were

randomly stacked in the kiln and stickered with aluminum strips. Specimens with thermocouples were placed in random locations within the stack. (Figure **2.4**) A heat treatment cycle was then initiated, achieving the time/temperature requirements as outlined in ISPM 15 standards. The core temperature of the thermocouple-containing specimen was monitored until 56 °C was reached, at which time 40 minutes were allowed to lapse, 10 minutes longer than accepted in the standard to account for variability in the test specimens, before removing the samples. Conditions upon opening the kiln were steamy, and samples were moist to the touch.

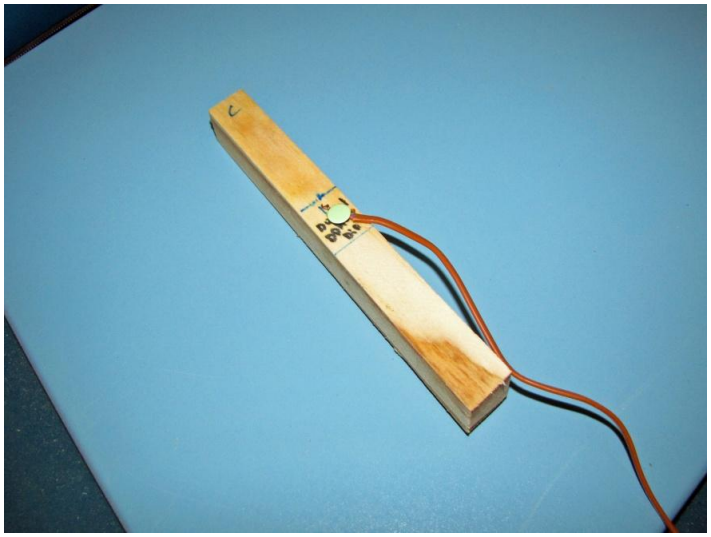

Figure 2.3

Thermocouple used to measure surface temperature, held by a push-pin.

Figure 2.4

Test specimens placed in kiln prior to HT.

The testing procedure used in the previous experiment by the author served as a guideline for the set-up of this study. 100 milliliters (ml.) of deionized water and 400 grams (g.) of sand were added and dispersed evenly to cover the bottom of non-sterile plastic containers measuring 12 in. x 9 in. x 5 in., (30.4 cm. x 22.8 cm. x 12.7 cm.), (L x W x D). Unseasoned pine boards were then cut from the 1 in. (2.5 cm.) stock of the milled trees and a single piece approximately 1 in. x 3 in. x 8 in., (2.54 cm. x 7.62 cm. x 20.32 cm.) (R x T x L) was then placed in each container in direct contact with the sand, oriented with the length parallel to the length of the container. The function of the non-sterile unseasoned pine was to serve as a uniform source of inocula beneath all test specimens. A single piece of screen mesh measuring 12 in. x 11 in., (30.4 cm. x 27.9 cm.), was then placed in the container above the unseasoned pine to separate the test samples approximately 1 in. (2.5 cm.) from the sand and pine board as shown in Figure **2.5**. The 5 replicates of a single treatment group were then placed in each container.

Lids were placed on the containers and the containers were placed in an environmental chamber at 85% relative humidity (RH) and 75 °F (23.8 °C) for 28 days. A visual examination was conducted after 7 days and then every 3 days for the remainder of the testing period to track initial mold growth on test specimens. After the 28 day testing period, each specimen was examined and evaluated for surface mold. A rating with a corresponding percent value as shown in Table **2.2** was given for both control and treated ends of each test specimen. Specimens remained in the non-sterile test chambers for 21 days prior to cleaning. Surface mold was removed using a nylon bristle brush and a mixture of deionized water with a mild detergent (Palmolive) as shown in Figure **2.6**. Each specimen was scrubbed with the detergent mixture, rinsed with deionized water, and patted dry with paper towels. As shown in Figure **2.7**, many samples, though cleaned, had extensive staining. Samples were returned to the environmental chamber for 24 hours prior to being subjected to a second HT. The sand from each testing container was removed.

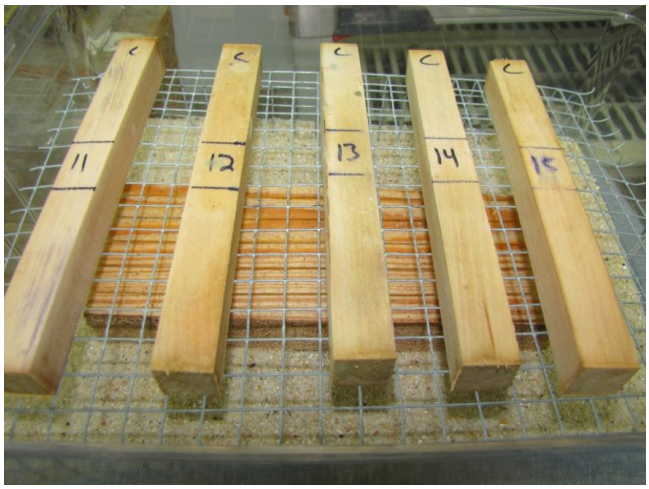

Figure 2.5

Sample placement for a single treatment group.

Table 2.2

Rating system for surface mold evaluation. Numbers are an assigned numeric value for analysis.

Rating	% Value
0	0
-	12.5
+	25
+-	37.5
++	50
++-	62.5
+++	75
+++-	87.5
++++	100

Figure 2.6

Sample cleaning with mild detergent.

Figure 2.7

Cleaned samples with stains on both treated and control ends.

Cycle 2

Test samples were subjected to a second HT using the same method outlined earlier. The collected sand from Cycle 1 was redistributed (400 g.) to each non-sterile testing container and 150 ml. of deionized water was added. The unseasoned pine board in each unsterile testing container was replaced with a fresh, unseasoned pine board. After HT, specimens were re-placed in the unsterile containers and placed in an environmental chamber at 85% relative humidity (RH) and 75 °F (23.8 °C) for 28 days. A visual examination was conducted after 7 days and then every 3-4 days for the remainder of the testing period to track initial mold growth on test samples. At the end of the testing period, samples were evaluated and rated for the presence of surface mold.

Results and Discussion

Cycle 1

Ambient core and surface temperatures of test specimens were approximately 25 °C (77 °F), prior to HT. Once the HT cycle was initiated, thermocouple readings for core temperature were monitored until 56 °C was reached. As shown in Figure **2.8**, the minimum core temperature was reached 30 minutes into the HT cycle. At the time a core temperature of 56 °C was achieved, surface temperature readings were approximately 3 °C higher. At the end of the required 30-minute HT cycle, the core temperature had reached 64 °C (147 °F) with surface temperatures approximately 1 °C higher, indicating the test specimens were nearing an equilibrated temperature.

Figure **2.9** shows the average day in which mold growth was evident on either treated or control ends of specimens per treatment group. As shown in the figure, most

control ends of test specimens had visible mold growth between day 7 and day 10. All control ends had visible mold growth by day 12, (an average of the 7, 10, and 13 day evaluations) the same time at which most treated ends had visible mold growth. The treatment that delayed visible mold growth for the longest amount of time was the dip treated 10% ai DOT + high concentration Si-Plus spray treatment with an average visible growth 20 days into the study.

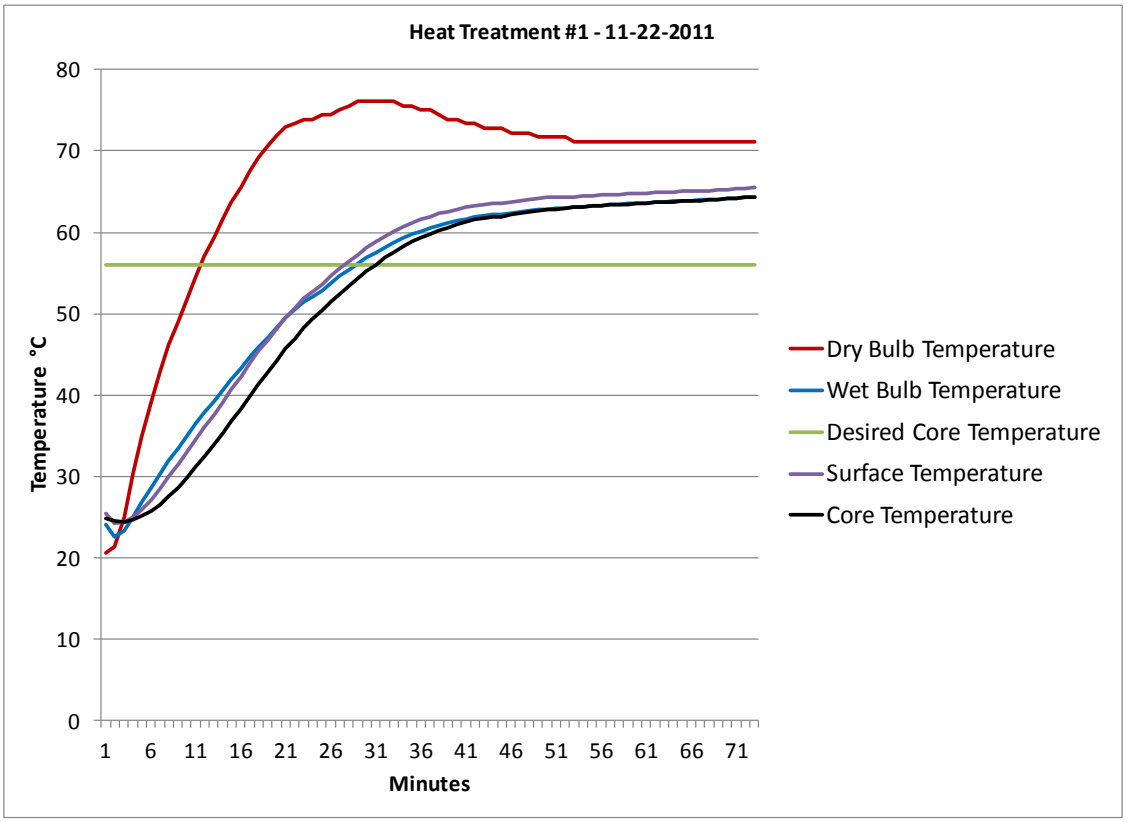

Figure 2.8

First HT of test specimens.

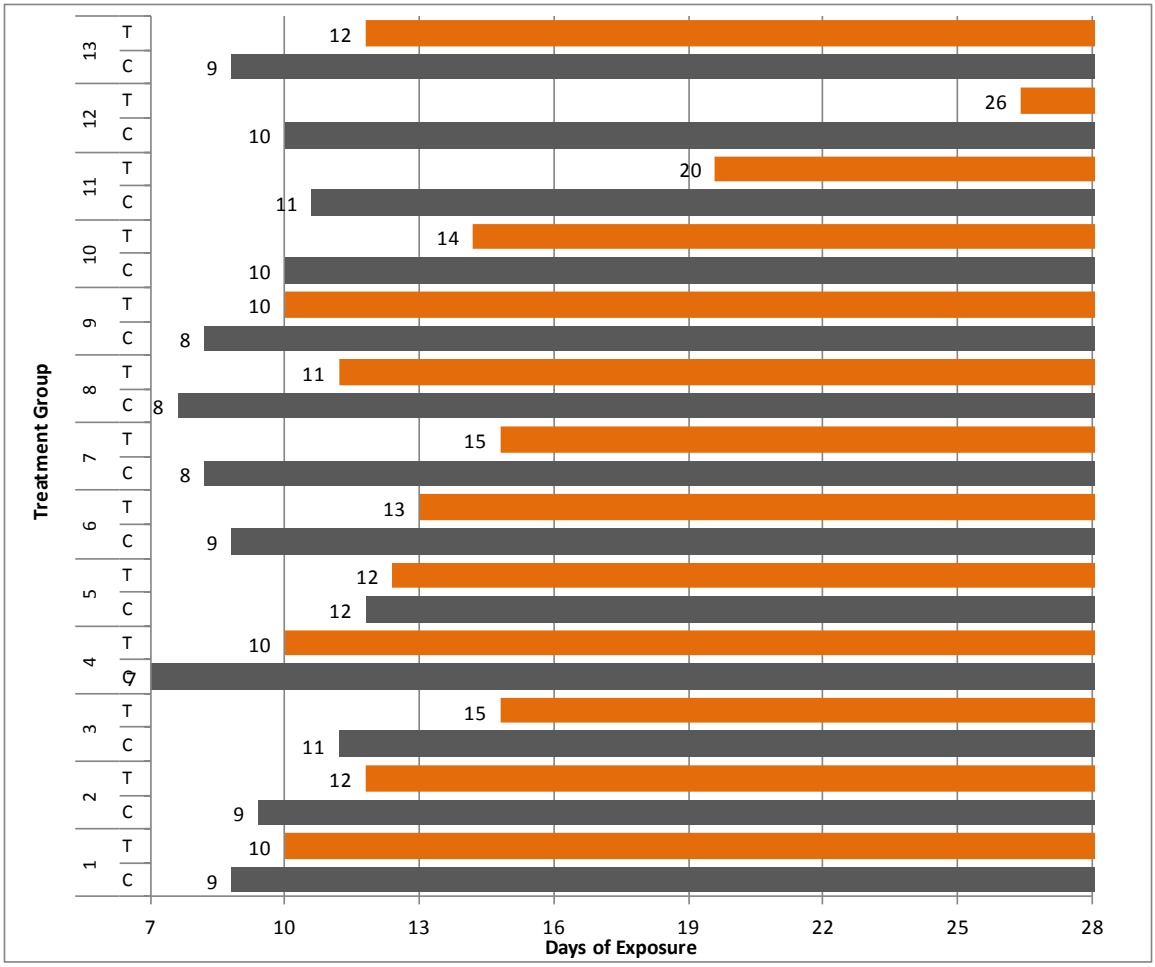

Figure 2.9

Average (5 replicates) time after HT that surface mold was observed on control and treated ends of test specimens during the first exposure cycle. Visual examinations were made every three days (after an initial 7 day examination) until the final evaluation. T = treated end, C = control end. Complete treatments given in Table 2.1.

Ratings were given to both control and treated ends of test samples and the difference was reported as the percent reduction in surface mold on treated ends (i.e., for each treatment group, the average treated rating was subtracted from the average control rating to determine the difference). Figures **2.10** – **2.15** show treatment groups that had at least 25% less surface mold on the treated ends than the control ends during the Cycle 1

exposure. Listed under each figure is the average rating for the control and treated ends expressed as a percent coverage of surface mold. The control rating minus the treated rating determines the percent reduction in surface mold for each treatment group. General appearance of the test specimens was not entirely indicative of the lack or presence of mold growth. The treated ends of many test specimens appeared to have little growth (no spores were present and thus very little color), but under close examination hyphal growth was often evident. It has been documented that some

"…chemicals may have either fungicidal and/or sporocidal properties (fungal hyphae and spores are killed), or fungistatic and/or sporostatic properties (hyphal growth and spore germination is retarded)" (Xiao and Kreber 1999).

Figure 2.10

Gum specimens dip treated with a 1.5% ai DDAC solution. Average control rating = 50 and average treated rating = 20 for the first exposure period.

Figure 2.11

Gum specimens spray treated with a low concentration Si-Plus solution. Average control rating = 80 and average treated rating = 23 for the first exposure period.

Figure 2.12

Gum specimens dip treated with a low concentration Si-Plus solution. Average control rating = 73 and average treated rating = 48 for the first exposure period.

Figure 2.13

Gum specimens spray treated with a high concentration Si-Plus solution. Average control rating = 83 and average treated rating = 40 for the first exposure period.

Figure 2.14

Gum specimens dip treated with a high concentration Si-Plus solution. Average control rating = 70 and average treated rating = 30 for the first exposure period.

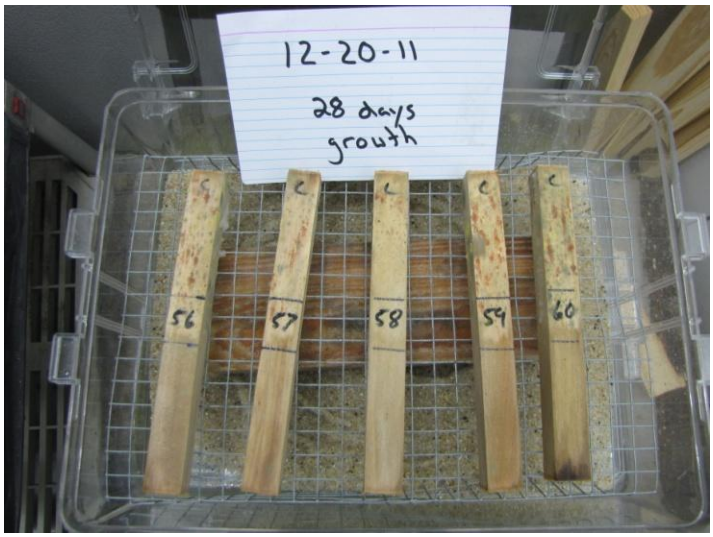

Figure 2.15

Gum specimens dip treated with a 10% ai DOT solution and spray treated with a high concentration Si-Plus solution. Average control rating = 83 and average treated rating = 10 for the first exposure period.

Figure **2.16** shows the average ratings for percent mold coverage given for each treatment group. Treatment groups are arranged in order of least percent surface mold on treated ends to greatest. As shown in the figure, both low and high concentration Si-Quat spray treatments, both low and high concentration Si-Quat dip treatments, and the 10% DOT dip treatment, had little to no affect on mold growth.

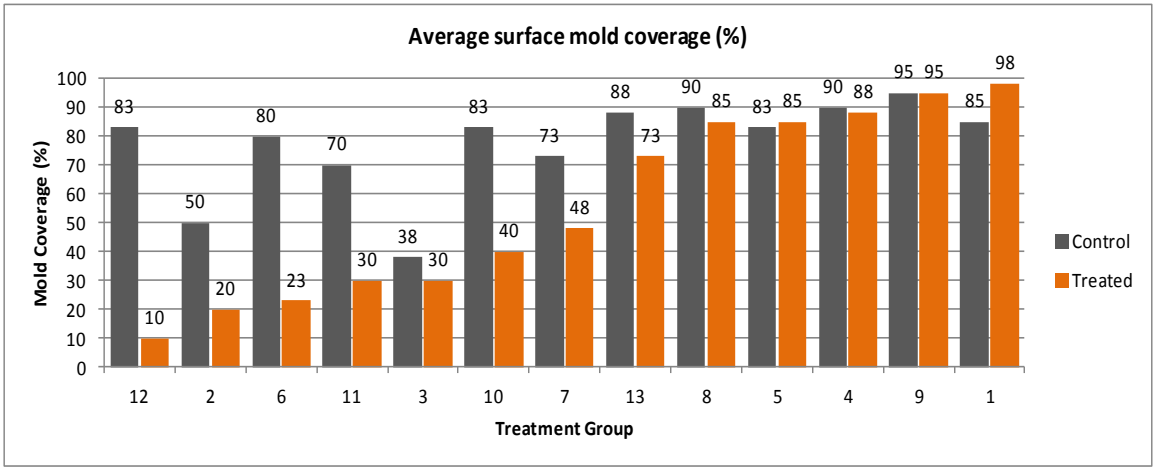

Figure 2.16

Average (5 replicates) percent coverage of mold growth on gum test specimens in Cycle 1 for control and treated ends per treatment group. Complete treatments are given in Table 2.1.

As shown in Figure **2.17**, all Si-Plus treatments exhibited at least 25% less surface mold than control ends. The average reduction in surface mold growth over control ends by the DOT + high concentration Si-Plus spray treatment was 73%. Spray treatments appeared to exhibit more control over surface mold growth than dip treatments for silane-based treatments. As shown in the figure, there was a significant gap between the moldicide control provided by Si-Plus and the control provided by Si-Quat. 1.5% DDAC solution applied by a dip application reduced surface mold on treated ends of gum test specimens by 30%. The low concentration Si-Quat dip treatment group and the 10% DOT dip treatment group are not shown in Figure **2.17** because surface mold growth on treated ends was higher than control ends.

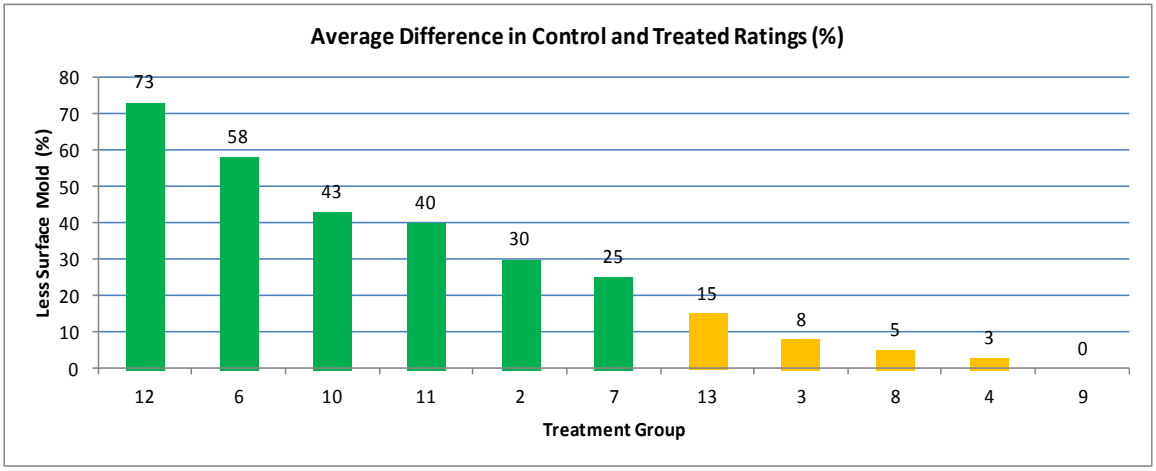

Figure 2.17

Average (5 replicates) percent difference in mold growth on treated and control ends of test specimens in Cycle 1. The percent difference is calculated by subtracting the average treated rating from the average control rating in each treatment group. Treatments shaded in green reduced surface mold by at least 25%. Complete treatments are given in Table 2.1.

Cycle 2

Ambient core and surface temperatures of test specimens were approximately 25 °C (77 °F), prior to the second HT. Once the HT cycle was initiated, a thermocouple reading for core temperature was monitored until 56 °C, the minimum core temperature as specified in ISPM 15, was reached. As shown in Figure **2.18**, the minimum core temperature was reached 30 minutes into the HT cycle. When the core temperature of the test specimen reached 56 °C, surface temperature readings were approximately 3 °C higher. At the end of the required 30-min. HT cycle, the core temperature had reached 64 °C (147 °F) with surface temperatures approximately 1 °C higher, indicating the test specimens were nearing an equilibrated temperature.

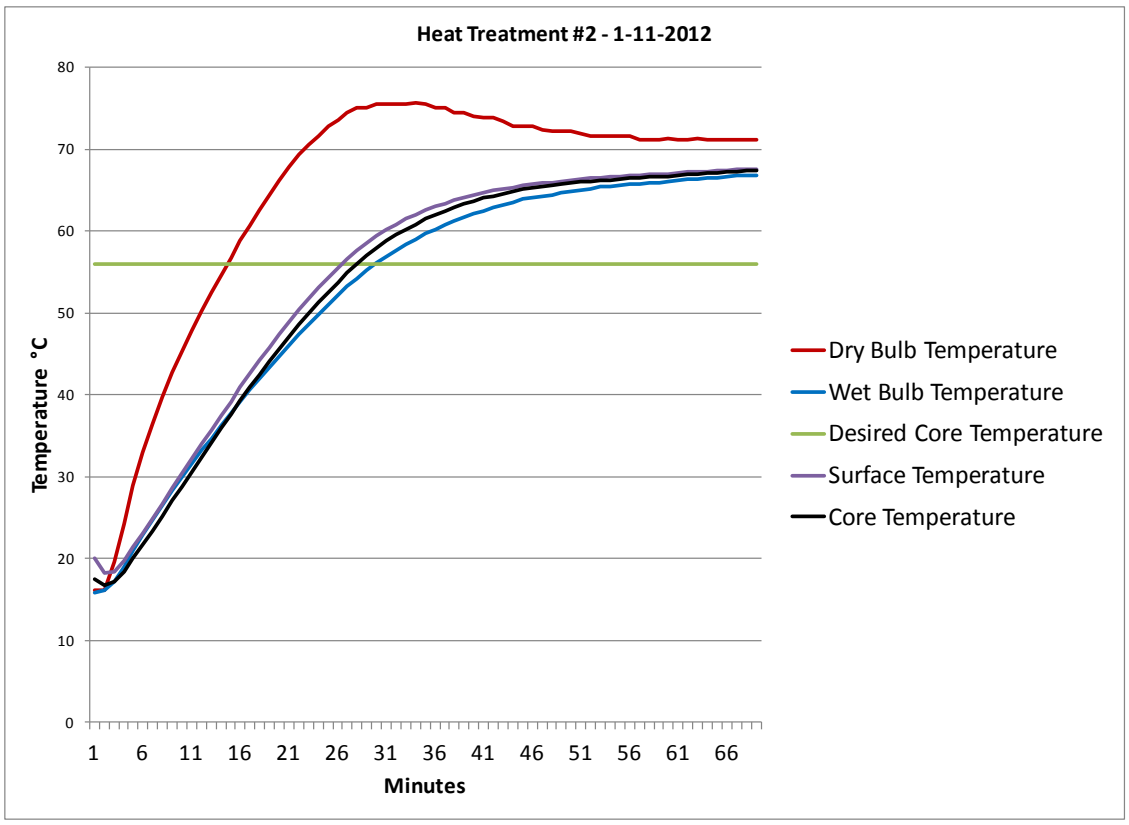

Figure 2.18

Second HT of test specimens.

Figure **2.19** shows the average day in which mold growth was evident on either treated or control ends per treatment group from the beginning of the exposure period. As shown in the figure, most control ends of test specimens had visible mold growth between day 7 and day 9. All control ends had visible mold growth by day 11, (an average of the 7, 10, and 13 day evaluations), with the exception of the control ends of the Boracare with Mold Care treatment group which did begin growth until around day 23. Most treated ends had visible mold growth between 7 and 12 days. Refer to Figures **2.20** – **2.24** for treatment groups that reduced surface mold by at least 25% during the Cycle 2 exposure.

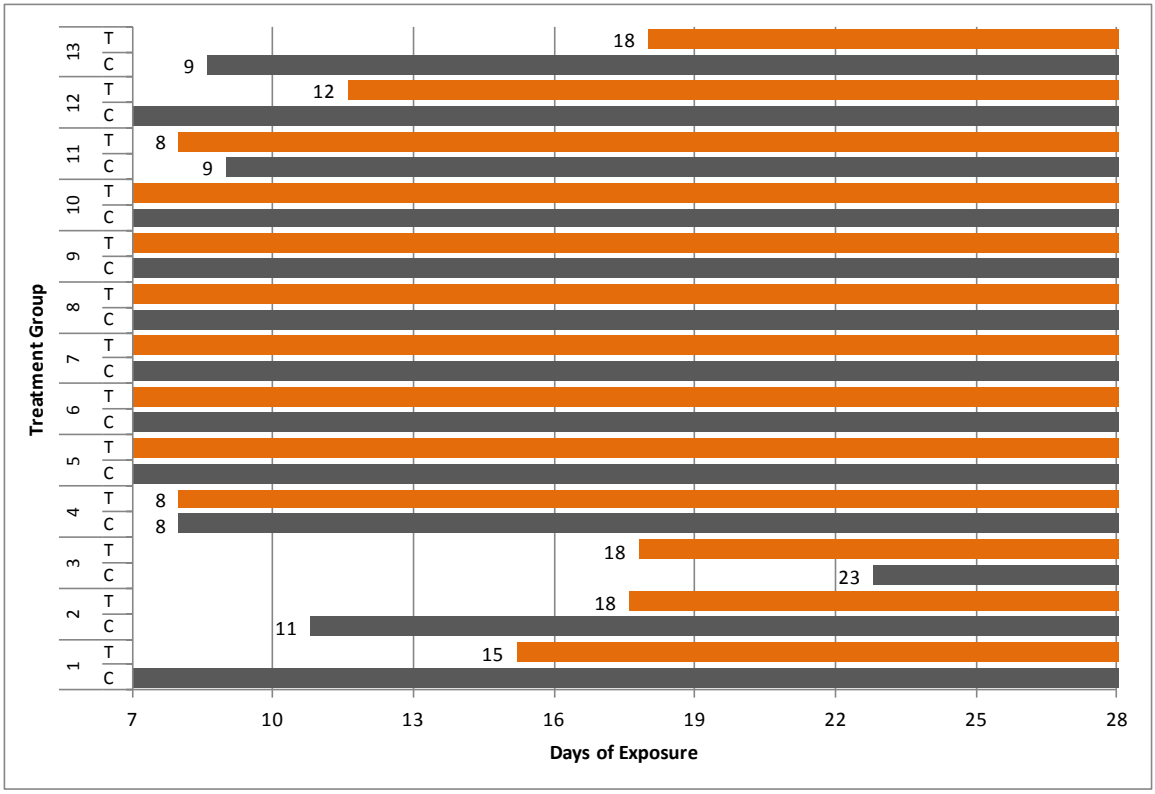

Figure 2.19

Average (5 replicates) time after HT that surface mold was observed on control and treated ends of test specimens during the second exposure cycle. Visual examinations were made every three days (after an initial 7 day examination) until the final evaluation. T = treated end, C = control end. Complete treatments given in Table 2.1.

Figure 2.20

Gum specimens dip treated with a 1.5% ai DDAC solution. Average control rating = 80 and average treated rating = 55 for the second exposure period.

Figure 2.21

Gum specimens spray treated with a low concentration Si-Plus solution. Average control rating = 85 and average treated rating = 53 for the second exposure period.

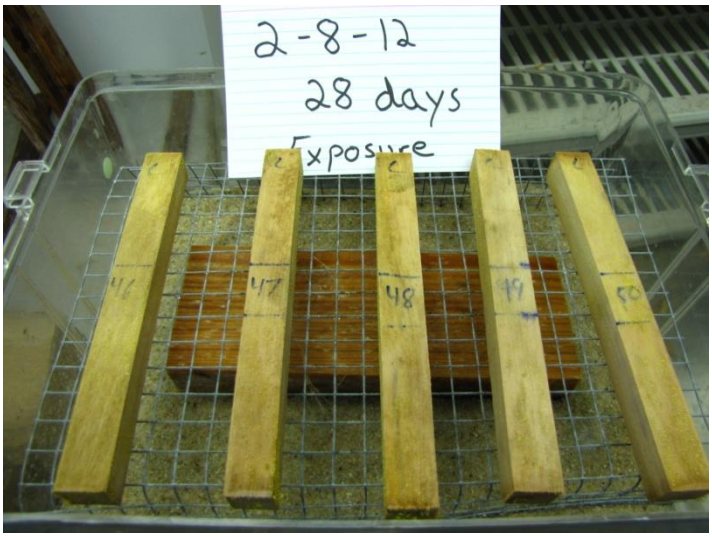

Figure 2.22

Gum specimens spray treated with a high concentration Si-Plus solution. Average control rating = 90 and average treated rating = 58 for the second exposure period.

Figure 2.23

Gum specimens dip treated with a high concentration Si-Plus solution. Average control rating = 93 and average treated rating = 65 for the second exposure period.

Figure 2.24

Gum specimens dip treated with a 10% ai DOT solution and spray treated with a high concentration Si-Quat solution. Average control rating = 65 and average treated rating = 33 for the second exposure period.

Figure **2.25** shows the average ratings for percent mold coverage given for each treatment group during the second exposure cycle. Treatment groups are arranged in order of least percent surface mold on treated ends to greatest. As shown in the figure, both low and high concentration Si-Quat spray treatments, both low and high concentration Si-Quat dip treatments, and the low concentration Si-Plus dip treatment, had little to no effect on mold growth during the second exposure period.

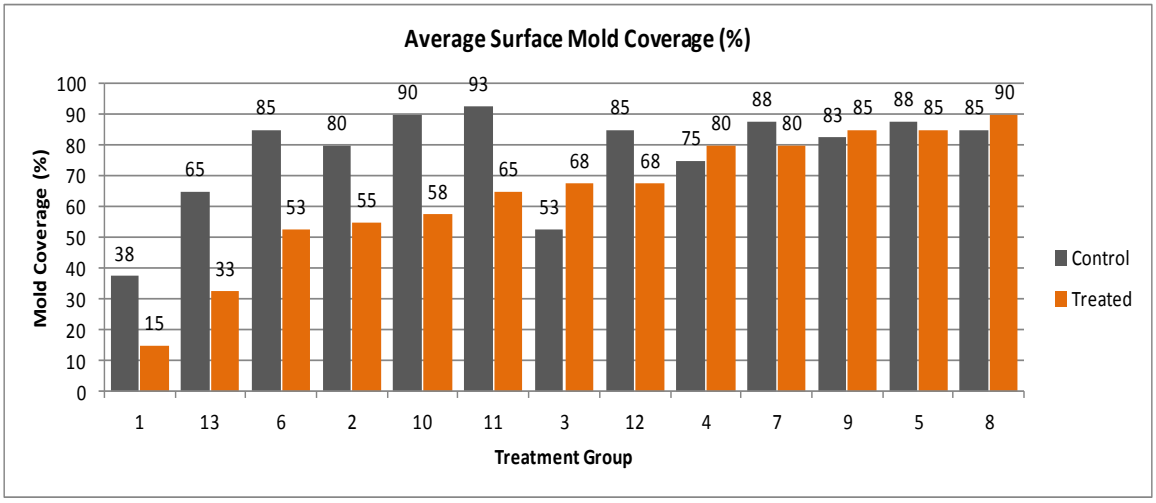

Figure 2.25

Average (5 replicates) percent coverage of mold growth on gum test specimens in Cycle 2 for control and treated ends per treatment group. Complete treatments given in Table 2.1.

As shown in Figure **2.26**, both low and high concentration Si-Plus spray treatments, the high concentration Si-Plus dip treatment, the DOT with high concentration Si-Quat spray treatment, and the DDAC dip treatment groups reduced surface mold growth by 25% or greater when compared to control end ratings during the second exposure period. The high concentration Si-Quat dip treatment, both low and high concentration Si-Quat spray treatments, and the Boracare with Mold Care treatment groups are not represented in this figure because surface mold growth on treated ends was higher than control ends.

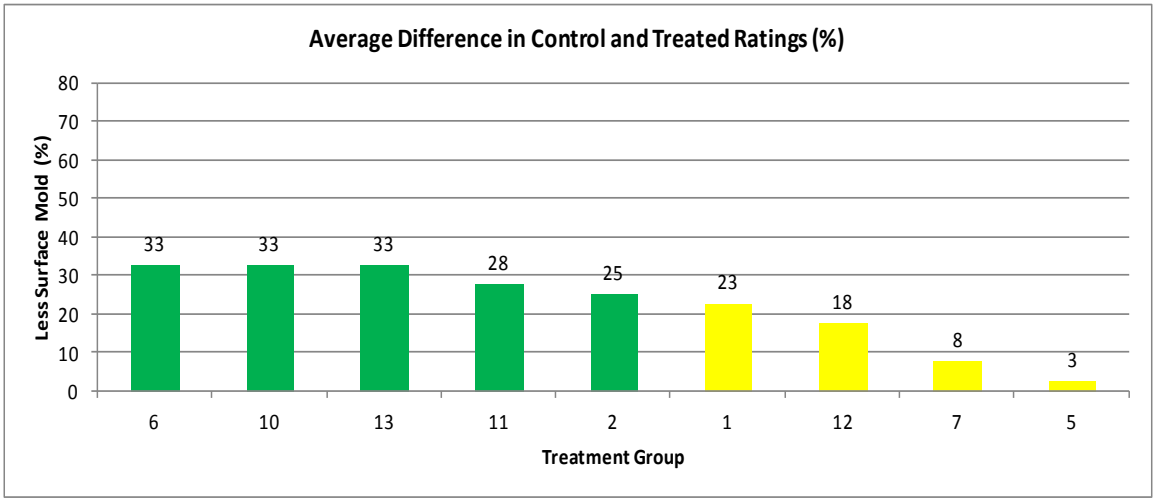

Figure 2.26

Average (5 replicates) percent difference in mold growth on treated and control ends of test specimens in Cycle 2. The percent difference is calculated by subtracting the average treated rating from the average control rating in each treatment group. Treatments shaded in green reduced surface mold by at least 25%. Complete treatments given in Table 2.1.

Overall Cycle

As shown in Figure **2.27**, variable results were obtained when comparing the percent difference in surface mold presence on treated and control ends of gum test specimens between Cycle 1 and Cycle 2. Four treatment groups; the 1.5% DDAC dip treatment, the low concentration Si-Plus spray treatment, the high concentration Si-Plus spray treatment, and the high concentration Si-Plus dip treatment, all maintained at least a 25% reduction in surface mold growth over control ends for both exposure cycles. The most significant reductions in mold growth occurred during the first exposure cycle for the low concentration Si-Plus spray treatment and the 10% DOT dip with a high concentration Si-Plus spray treatment. Three treatment groups had greater reductions of surface mold growth in Cycle 2 than in Cycle 1.

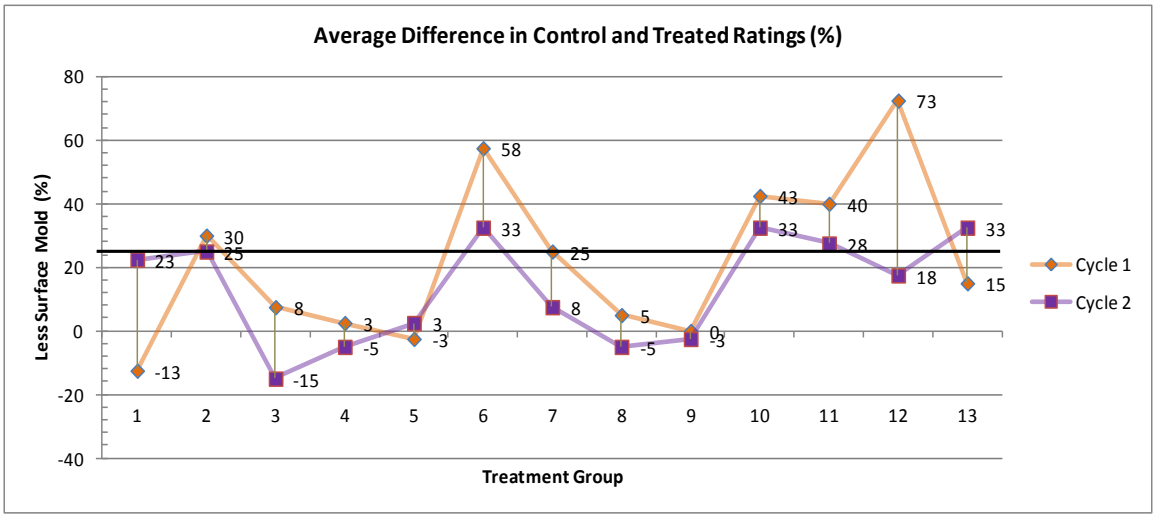

Figure 2.27

Comparison of the average percent difference in surface mold between control and treated ends of test specimens for each treatment group for Cycle 1 and Cycle 2 evaluations. Complete treatements are given in Table 2.1.

Conclusion and Recommendations

Results of this study utilizing 3-(trimethoxysilyl) propyldimethyloctadecyl ammonium chloride to inhibit surface mold on gum test specimens in conjunction with standardized heat treatments indicate that when reinforced with the proprietary additive (Si-Plus) by the manufacturer it can reduce the presence of surface mold by 25% or greater in a 28 day test cycle. Results do not support Si-Quat as a solitary treatment method by either dip or spray but further testing should be conducting utilizing Si-Quat in conjunction with DOT. Results of this testing procedure support the efficacy of didecyl-dimethylammonium chloride (DDAC), a chemical utilized extensively in current mold control products. However, should a second cleaning and third exposure cycle be conducted, a trend might be observed indicating a loss in efficacy.

Results of the current test procedure are not strictly comparable to results of the previous experiment by the author. Boracare® with Mold Care® performed significantly better in the previous experiment which utilized both gum and pine test specimens. Age of the chemical or possible insufficient mixing of the chemical could be factors in the varying results. Also, the pine boards utilized as a source of inocula beneath the test specimens grew little to no mold during either test cycle, though staining did occur. In the previous experiment significant growth was observed on the pine boards. Perhaps the handling process of the raw material from forest to laboratory did not allow sufficient exposure to mold spores in the environment, thus retarding mold growth.

It is recommended that the testing protocol be tested further and standardized to yield consistent results. Observations for mold appearance during the testing cycle indicate that test containers do not support mold growth at uniform times. A testing container large enough to hold all test specimens being evaluated is recommended to the protocol to ensure a uniform source of inoculum to all test specimens.

CHAPTER III

OBSERVED COLOR PHENOMENA AND BEHAVIORAL ABNORMALITIES OF *RETICULITERMES* SP. IN AWPA E1-09 STANDARD LABORATORY TERMITE TEST

Introduction/Literature Review

Laboratory termite tests of treated or non-treated wood materials primarily focus on either termite feeding or repellency. One such procedure for testing is the American Wood Protection Association E1-09 Standard Method for Laboratory Evaluation to Determine Resistance to Subterranean Termites (AWPA 2010A). With this procedure, weight losses of wood wafers exposed to worker termites in non-sterile test chambers are used as the basis of termite resistance. However, tests have indicated that factors influencing termite behavior, other than attracting or repelling, may affect the results of the test. For example, interactions between termites and bacteria and/or fungi have been shown to affect termite feeding (Amburgey 1977; Cornelius 2002A, *et al*. 2002B). While determining the termite resistance of organosilane-treated wood wafers was the primary objective of this study, the behavioral pattern of termites during this test was also observed and reported.

A standard laboratory termite test was conducted in September of 2010 using termites from a single colony of *Reticulitermes* sp., gathered in East Central Mississippi. Testing was performed adhering to procedures outlined in the AWPA E1-09 Standard

termite test. Wood wafers used in the test were treated with an organosilane compound. Observations of worker termites exposed to treated wood wafers during testing included disoriented, convulsive type movements as well as sluggish behavior. *Post mortem* observations indicated that some worker termites exposed to treated wood wafers showed a light pinkish to red color, primarily in the head area extending to the abdomen. The abnormal behavior and *post mortem* color phenomena observed in this test resemble observations in past studies on the association between termites and the bacterium, *Serratia marcescens* (Bizio).

Experimental

AWPA E1-09: Standard Method for Laboratory Evaluation to Determine Resistance to Subterranean Termites was the protocol for this experiment (AWPA 2010A) Adhering to the standard, clear sapwood pine wafers measuring 1.00 in. x 1.00 in. x 0.25 in. (25 mm. x 25 mm. x 6 mm.), (R x T x L), with 4-6 growth rings per inch were obtained from a single, seasoned parent board. Both no-choice and choice testing procedures were evaluated. For each treatment group, five test wafers were evaluated. Both no-choice and choice tests evaluated four treatment groups, plus a control group, consisting of non-treated pine sapwood wafers. Wafers were treated at two concentration levels with the organosilane 3 (trimethoxysilyl) propyldimethyloctadecyl ammonium chloride, (Si-Quat), by both dip and vacuum application methods. The Si-Quat used in this study is widely known for its antimicrobial abilities (Isquith *et al*. 1972; Hayes & White 1984; Kemper *et al*. 2005; Monticello *et al*. 2009). Dip treatments were made by momentarily immersing each wafer in the desired solution. Vacuum treatments were

done by submerging wafers in solution and placing under vacuum at 27 in. (68.6 cm.) Hg for 15 minutes. Treated test samples were then conditioned at 50 °C (122 °F) until a constant weight was reached.

Termites (*Reticulitermes* sp.) were collected from a pine log on the Mississippi State University Dorman Lake Test Site in AWPA Hazard Zone 4 (Figure **3.1**). Since *Reticulitermes flavipes* (Kollar) is the primary termite species at this site, test organisms are believed to be *R. flavipes*. However, the termites were not specifically identified to species. For no-choice testing, non-sterile test containers were prepared by adding 150 g. of sand and 20 ml. (deviation from the standard 30 ml.) of deionized water. For choice testing, non-sterile test containers were prepared by adding 300 g. of sand and 40 ml. of deionized water. A single wafer was placed in direct contact with the moistened sand in no-choice test containers and 1 g. of termites were weighed and added to each container. Two wafers, one treated and one control, were placed in direct contact with the moistened sand on opposing ends of the choice test containers and 1 g. of termites were added to each container. Lids were loosely fitted to each container. The duration of this test was twenty-eight days, during which time termite feeding behavior was observed and *post mortem* observations were made.

Testing containers were evaluated for termite mortality and test wafers were rated for termite attack as outlined in AWPA standard E1-09. Block m0ass loss results for the testing procedures were recorded and mean values were analyzed by Tukey, a test for the significance of means.

Figure 3.1

Collection site of *Reticulitermes* sp. for testing.

Results and Discussion

Observations

Early in the testing period, termites exposed to wood wafers treated with both low (1% ai) and high (2.5% ai) concentrations of the Si-Quat exhibited erratic behavior. Movements were hyper-active and convulsive, resembling spasms. As the study progressed, termites adopted an increasingly sluggish, lethargic behavior to the point that mortality was believed to have been reached. This observation was nullified when abrupt disturbance of the test chambers prompted movement from the termites. A pinkish coloration on deceased termites was observed in various test chambers, but this coloration was not evident on living termites. Pink stains remained on the sand in the containers, evidently where the termites deteriorated with time. At the end of the testing period, some termites exhibiting the pink coloration were removed for analysis and either

placed in 95% ethyl alcohol or frozen for preservation. Specimens in the alcohol solution lost the coloration within twenty-four hours, while frozen specimens retained the color (Figures **3.2** and **3.3**). These observations prompted further investigation. No unusual observations were made regarding worker termites exposed to untreated wood wafers.

Figure 3.2

Frozen specimen. Varying intensities of pigment noted in the head, abdomen, and legs.

Figure 3.3

Frozen specimen. Note colorations on the head and legs.

In a test reported in 1939, strains of bacteria were isolated and analyzed because of their observed ability to kill termites (DeBach and McOmie 1939). The author reported symptoms of termites infected with one strain, *Serratia marcescens* (Bizio), that included observations similar to those noted in the presented study and was believed to be the first such observation on record for the Order Isoptera. DeBach and McOmie reported varying red colorations on termites *post mortem* that affected, and was most pronounced, in the head. They also noted the coloration in other anatomical areas such as legs, thorax, etc. The coloration was not observed prior to death, but characteristics of the infected living termites included lethargy, no feeding activities, isolation from other termites, and a loss of motion leading up to death. They hypothesized that the isolation observed could, in part, be due to hypersensitivity, and that *post mortem* movements observed, were likely due to effects on the nervous system.

Subsequent studies with *S. marcescens* have indicated that it is a facultative anaerobe that is a symbiont of the anaerobic protozoa in the gut of termites (Adams and Boopathy 2005). Although the termites ingest wood, it is their symbiotic protozoa that digest the wood components into compounds that serve as food for the anaerobic protozoa, their symbiotic bacteria (e.g., *S. marcescens*), and the termites (Adams and Boopathy 2005; Brune and Friedrich 2000). *S. marcescens*, in turn, scavenges oxygen coming through the termite gut walls to use for its respiration and to keep the gut anaerobic, as required by the protozoa (Adams and Boopathy 2005). When something disrupts these symbiotic relationships (*e.g*., termites treated with immuno-suppressing compounds) the termites' immune defense response is suppressed (Connick *et al*. 2001; Osbrink *et al*. 2001). The increasing stress on the termites is believed to be associated with triggering the symbiont *S. marcescens* to become pathogenic. This, rather than insecticidal toxicity, may be the mode of action of some wood preservatives with observed termiticidal toxicity. The organosilane used to treat wood specimens in the current study may have this mode of action.

Isolated strains of *S. marcescens* were used in a study on *Coptotermes formosanus* (Shiraki) (Connick *et al.* 2001). In this test, termites exposed to *S. marcescens* had high mortality rates, and red features were observed on the termites *post mortem*, as was observed in the present study. *S. marcescens* has been used as a biological indicator of the presence of organisms that have made contact with the bacterium because of its ability to be monitored based on its distinctive pigment which ranges from red to light pink (Yu 1979). The pathogenicity of *S. marcescens* has been documented for not only termites but many mammals, as well as a causal agent in many respiratory, urinary and

musculoskeletal infections (Rosahn and Hu 1933; Kahn *et al*. 1977; Yu 1979). Particularly of interest to the present study is that infection by the bacterium is a common risk associated with taking antimicrobial medicines (Yu 1979). This may be correlated with the use of an antimicrobial, Si-Quat, treatment in the present study. Also of interest is the pigment's confirmed solubility in alcohol and light sensitivity (DeBach and McOmie 1939; Yu 1979). These variables are consistent with observations in the present study in which the pigment was lost from termite specimens placed in an alcohol solution as well as fading of the pigment while photographs were being taken, possibly due to the intense, direct lighting used.

No-Choice Test Procedure

The percent mass loss for each treatment group was evaluated and as shown in Figure **3.4**, higher concentration treatment groups had significantly less mass loss, according to a Tukey test. All treatment groups were significantly different than the control group. As shown in Figure **3.5**, the average percent mass loss had a negative correlation with the retention ai of each treatment group, decreasing as retention increased, however, mass loss was insignificant between the vacuum treated groups. The average block rating and termite mortality for each treatment group is presented in Table **3.1,** followed by the cleaned samples after testing, arranged by treatment group in Figure **3.6**.

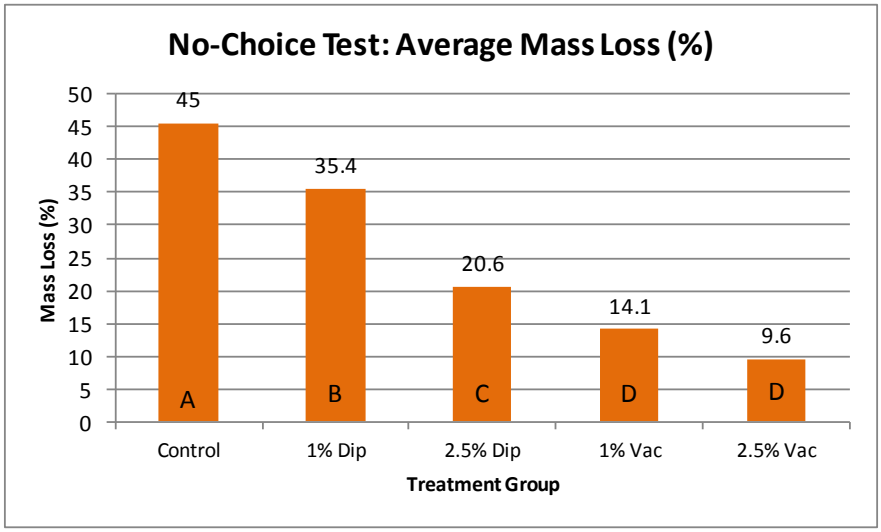

Figure 3.4

Average (5 replicates) percent mass loss for each treatment group in the no-choice testing procedure. Treatments with the same letter are not significantly different.

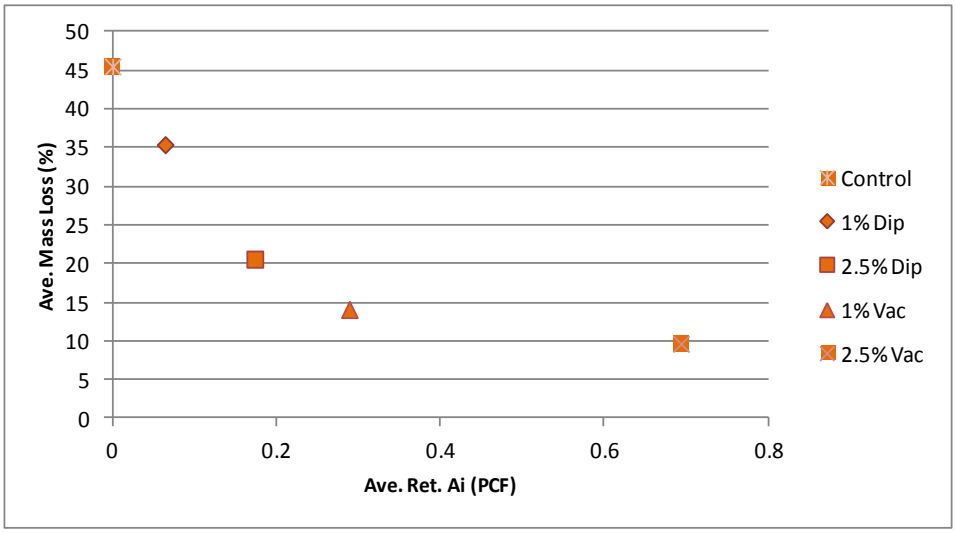

Figure 3.5

Average (5 replicates) percent mass loss in correlation with group treatment levels, expressed as retention ai (pcf).

Table 3.1

Average (5 replicates) retention ai, mass loss percent, block rating, and termite mortality for each treatment group in the no-choice testing procedure.

	Treatment Group	Reps	Retention ai. (pcf)	Mass Loss (%)	Block Rating[A]	Mortality[B]
No-Choice	Untreated Control	5	---	45	0	S
	1% Dip	5	0.06	35.4	4	S
	1% Vacuum	5	0.29	14.1	7.2	C
	2.5% Dip	5	0.17	20.6	7.2	H-C
	2.5% Vacuum	5	0.69	9.6	8.8	C

[A] Block ratings were given subjectively following the procedures outlined in AWPA standard E1-09 and are expressed here as the average of 5 replicates per treatment group.

[B] Termite mortality was quantified as outlined in ASTM standard D3345-74. S = Slight (0%-33%), M = Moderate (34%-66%), H = Heavy (67%-99%), C = Complete (100%).

Figure 3.6

Wafers utilized in the no-choice testing procedure. Treatment groups are arranged vertically.

Choice Test Procedure

The percent mass loss for both the control and treated wafers for each treatment group was evaluated for termite preference within each treatment group and reported. As shown in Figure **3.7**, according to a Tukey test, for a 1% Si-Quat dip, the mass loss for treated wafers was not significantly different than that of control wafers. For each of the three remaining treatment groups, 2.5% Si-Quat dip, 2.5% Si-Quat vacuum, and 1% Si-Quat vacuum, the mass loss for control wafers was significantly greater than that of treated wafers, indicating termite preference to non-treated control wafers. The average block rating and termite mortality for each treatment group is presented in Table **3.2** followed by the cleaned wafers after testing arranged by treatment group in Figures **3.8 – 3.11**.

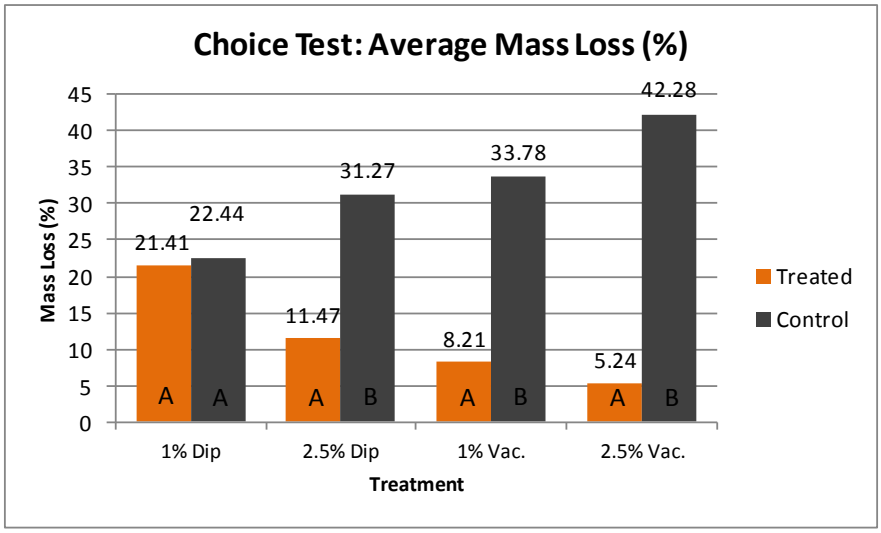

Figure 3.7

Average (5 replicates) percent mass loss for control and treated wafers by treatment group in the choice testing procedure. Treatments with the same letter are not significantly different.

Table 3.2

Average (5 replicates) retention ai, mass loss percent, block rating, and termite mortality for each treatment group in the choice testing procedure.

	Treatment Group	Reps	Retention ai. (pcf)	Control Mass Loss (%)	Control Block Rating[A]	Treated Mass Loss (%)	Treated Block Rating[A]	Mortality[B]
Choice	1% Dip	5	0.05	22.4	6.0	21.4	6.4	S
	1% Vacuum	5	0.27	33.8	0.8	8.2	9	S
	2.5% Dip	5	0.19	31.3	1.6	11.5	8.6	S
	2.5% Vacuum	5	0.71	42.3	5.2	5.2	9.5	S

[A] Block ratings were given subjectively following the procedures outlined in AWPA standard E1-09 and are expressed here as the average of 5 replicates per treatment group.
[B] Termite mortality was quantified as outlined in ASTM standard D3345-74. [S = Slight (0%-33%), M = Moderate (34%-66%), H = Heavy 67%-99%), C = Complete (100%)].

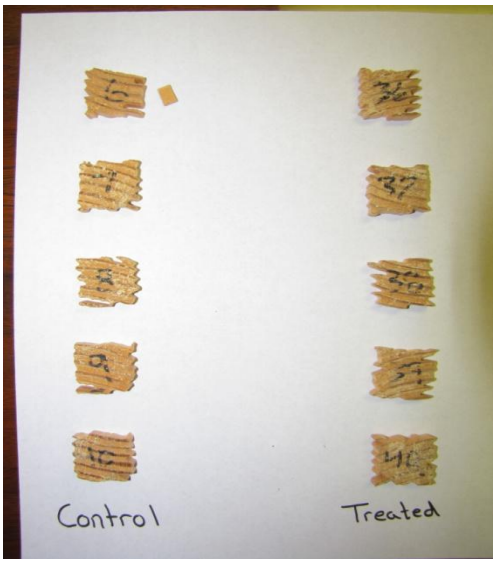

Figure 3.8

Paired control and treated wafers for the 1% ai Si-Quat dip treatment group in choice testing.

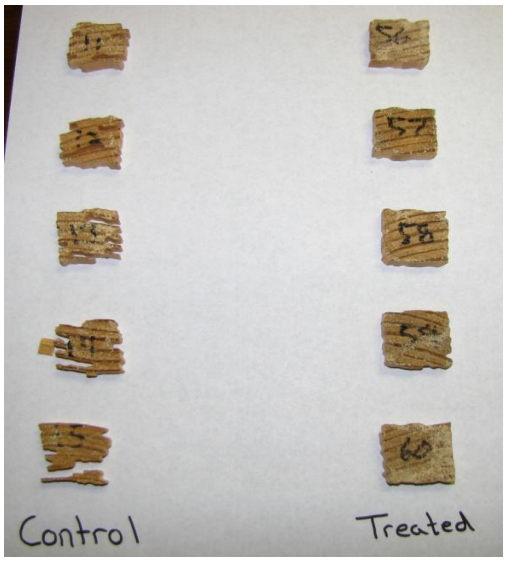

Figure 3.9

Paired control and treated wafers for the 2.5% ai Si-Quat dip treatment group in choice testing.

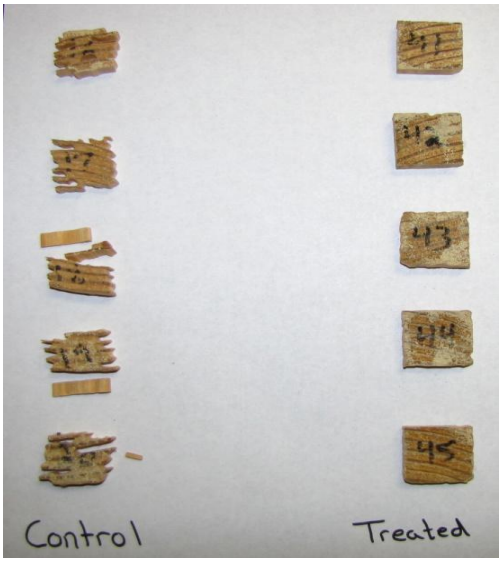

Figure 3.10

Paired control and treated wafers for the 1% ai Si-Quat vacuum treatment group in choice testing.

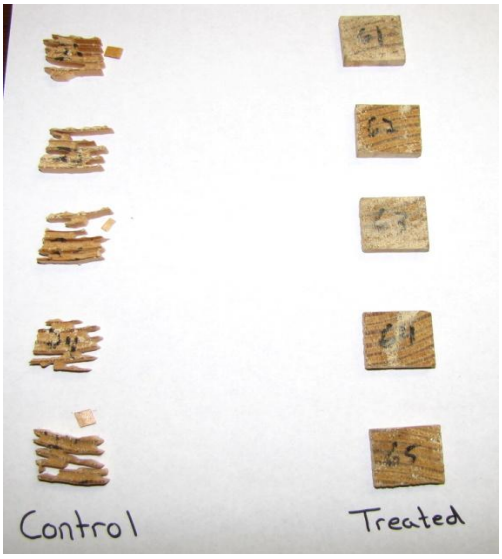

Figure 3.11

Paired control and treated wafers for the 2.5% ai Si-Quat vacuum treatment group in choice testing.

Conclusion and Recommendations

The primary goal of the present study was to examine feeding of termites on organosilane-treated wood wafers. Observations of worker termite behavior and *post mortem* appearance in the present study are similar to previously published studies in which a bacterium, *S. marcescens*, was isolated and exposed to termites under various experimental conditions. The observations in the present study were secondary to the goal of the study, and were unexpected, requiring further research. Under consideration is whether the pathogenic response of *S. marcescens* was selectively stimulated by the use of the Si-Quat treatment. Further research will be conducted to determine if the phenomena observed in the present study can be replicated on termites collected from the same colony. The termites will also be monitored more stringently and a larger sample group will be maintained for bacteria isolation. This work will be important for insuring

a healthy termite population when utilized in future basic and standardized testing procedures. Perhaps observations of termite behavior, and development of red coloration in dead termites, should be added to the protocol of AWPA Standard E1-09.

Further studies should be conducted to see if similar results are achieved on other wood destroying insects that contain *S. marcescens* internally. Significant feeding differences were obtained for both no-choice and choice testing procedures. Treated test wafers had significantly less percent mass loss as well as increased termite mortality over control test wafers in no-choice testing. For choice testing, higher percent active solutions as well as increased treatment retentions in vacuum treatments of treated wafers had significantly less percent mass loss than control wafers indicating preference for the untreated controls. Further testing should be conducted to determine the toxic thresholds for both dip and vacuum treatments with the organosilane, 3 (trimethoxysilyl) propyldimethyloctadecyl ammonium chloride.

CHAPTER IV

EVALUATION OF THE QUATERNARY AMMONIUM CHLORIDE 3-(TRIMETHOXYSILYL) PROPYLDIMETHYL OCTADECYL AMMONIUM CHLORIDE (SI-QUAT) AS A WATER REPELLENT ON SOUTHERN YELLOW PINE

Introduction/Literature Review

The interaction between wood and water has long been a facet of scientific study. Wood is a hygroscopic, or water-loving, material which is capable of absorption or desorption of water molecules through hydrogen bonding to maintain moisture equilibrium with its surroundings (Rowell and Banks 1985). The current moisture level in a given wood material can be measured and expressed as the percent moisture content (MC). The increase and decrease of moisture levels in wood material up to 30% MC, known as the fiber saturation point, can affect the dimensional properties of wood materials by causing wood to shrink and swell (Forest Products Laboratory 1999; Bowyer *et al*. 2003). Moisture level changes above the fiber saturation point, however, are insignificant to changes in dimension. When the MC of wood is above 19-20%, wood is susceptible to attack and degradation by many biological organisms, including fungi and wood destroying insects (Forest Products Laboratory 1999; Micales-Glaeser *et al*. 2004). Once wood has been dried, usually by a kiln or air drying process, to a moisture content below 19%, its susceptibility to degradation by these organisms is reduced; however, if

dried wood is utilized in a high moisture area where it will equilibrate to a higher moisture content, degradation is likely to occur.

Chemical treatments with the ability to inhibit or repel moisture from absorption into wood materials are currently available for use in the wood industry. The primary function of these water repellent treatments is to lessen the rate at which liquid or vapor water is absorbed into wood and is typically accomplished by the inclusion of a wax in the treatment formulation, where no chemical bonding actually exists between the treatment and wood substrate (Rowell and Banks 1985; Williams and Feist 1999). The silane compound 3 (trimethoxysilyl) propyldimethyloctadecyl ammonium chloride is a non-leaching antimicrobial which cures to substrate surfaces by covalent bonding (Isquith *et al*. 1972; Walters *et al.* 1973; Speier and Malek 1981; Monticello *et al*. 2009). There has been much research on the treatment of wood with silane compounds for dimensional stability because of their water repellent properties. Published data of this research is primarily focused on the hydrolysis of silanes which, once condensed, create three dimensional units in a process known as sol-gel. When certain silane compounds are impregnated into a wood substrate, the cell walls of the wood become "bulked" with these three dimensional units created by an interaction of the silane and the water within the wood cells (Donath *et al*. 2006A). Results of studies where wood was treated with silanes have shown improved dimensional stability and high water repellency imparted on wood materials (Donath *et al*. 2004; *et al*. 2006A; *et al*. 2006B, *et al*. 2007). The focus of many studies conducted utilizing silanes as wood treatments has been to evaluate the efficacies of silanes, impregnated either non-hydrolyzed (undergoing sol-gel once a reaction with the free and bound water in the wood cells has occurred) or as a sol

(prematurely hydrolyzed by a reaction with water prior to impregnation into the wood substrate), as water repellents or antimicrobials.

The purpose of this study is to evaluate the efficacy of the silane 3-(trimethoxysilyl) propyldimethyl octadecyl ammonium chloride as a water repellent on wood substrates applied as a sol (pre-hydrolyzed by dilution in water) using non-pressurized treatment methods.

Experimental

American Wood Protection Association standard E4-03 was used to model this experiment (AWPA 2007). Adhering to the standard, clear, flat-sawn pine wafers measuring 0.25 in. x 1.0 in. x 2.0 in. (0.64 cm. x 2.54 cm. x 5.08 cm.) (L x R x T) were cut from three individual, seasoned, boards. Each treatment group consisted of nine wafers; three wafers from each parent board. The samples were equilibrated at 60% RH and 75 °F (23.9 °C) (a deviation from the standard; 65% RH and 80 °F, 26.7 °C) (10.9% MC) in a non-sterile environmental chamber until a constant weight was reached (Figure **4.1**). The longitudinal, radial, and tangential dimensions were recorded for each test specimen using a digital caliper to calculate volumes to be used in determining solution retention for treatment. Twenty-four treatment groups were evaluated including four untreated control groups. Both spray and dip application methods were evaluated for each treatment (a deviation from the standard; no pressure treatment was evaluated), as well as oven-cured after treatment and non oven-cured after treatment as shown in **Table 4.1**. Chemical treatments evaluated in this study consisted of 3-trimethoxysilyl propyldimethyl octadecyl ammonium chloride (Si-Quat), Si-Plus, a proprietary

formulation with the same active ingredient as Si-Quat, and Thompson's® Water Seal®. The Si-Quat and Si-Plus products were provided by the chemical manufacturer and the Thompson's® Water Seal® was procured from a local retail store.

Figure 4.1

Pine wafers in conditioning chamber prior to testing.

Table 4.1

Treatment groups evaluated in testing, percent active ingredient of solutions used, target milligrams of active ingredient per square foot (mg ai/sqft), and the actual mg ai/sqft.

#	Treatment Group[A]	% ai[B]	Target mg ai/sqft[C]	Actual mg ai/sqft[D]
1	Low Conc. Si-Quat Spray	3.60	100	127
2	Low Conc. Si-Quat Spray*	3.60	100	132
3	Low Conc.Si-Quat Dip	0.51	100	100
4	Low Conc. Si-Quat Dip*	0.51	100	108
5	Low Conc. Si-Plus Spray	2.00	100	105
6	Low Conc. Si-Plus Spray*	2.00	100	106
7	Low Conc. Si-Plus Dip	0.51	100	88
8	Low Conc. Si-Plus Dip*	0.51	100	102
9	High Conc. Si-Quat Spray	5.40	150	207
10	High Conc. Si-Quat Spray*	5.40	150	168
11	High Conc. Si-Quat Dip	0.76	150	156
12	High Conc. Si-Quat Dip*	0.76	150	150
13	High Conc. Si-Plus Spray	2.00	150	161
14	High Conc. Si-Plus Spray*	2.00	150	164
15	High Conc. Si-Plus Dip	0.76	150	155
16	High Conc. Si-Plus Dip*	0.76	150	144
17	Thompson's® Water Seal® Spray	RTU	----	----
18	Thompson's® Water Seal® Spray*	RTU	----	----
19	Thompson's® Water Seal® Dip	RTU	----	----
20	Thompson's® Water Seal® Dip*	RTU	----	----
21	Water Treated Control Dip	NONE	----	----
22	Water Treated Control Dip*	NONE	----	----
23	Water Treated Control Spray	NONE	----	----
24	Water Treated Control Spray*	NONE	----	----

[A] Si-Quat = 3 trimethoxysilyl propyldimethyl octadecyl ammonium chloride in a 42% ai methanol solution diluted to final treatment solution with deionized water. Si-Plus = Si-Quat with proprietary additive in a 2% ai solution. Thompson's® Water Seal® = Ready to Use product. Control groups = Treated with deionized water, there is a control group for each method; dip, dip oven-cured, spray, and spray oven cured.

[B] Solutions were mixed on a wt/wt basis.

[C] Si-Quat manufacturer recommended treatment level.

[D] Actual Treatment Level

* Treatment groups were oven-cured after treatment.

Sample Treatment

Once test samples were equilibrated to a constant weight, the four untreated control groups were treated with deionized water. Control specimens receiving a dip treatment were submerged in deionized water momentarily and removed. Control specimens receiving a spray treatment were sprayed with deionized water using a Vaper Gravity Feed HVLP spray gun. The manufacturer of the Si-Quat and Si-Plus formulations recommended oven-curing treated substrates at 100 °C (212 °F) for 30 minutes to promote the efficacy of the chemical, as well as applying 100 milligrams of active ingredient per square foot (mg. ai/sqft) or 150 mg. ai/sqft. Both an oven-cured control group and non oven-cured control group were used in this study for each treatment method, dip and spray, per the manufacturer's recommendation for direct comparison with oven-cured and non oven-cured treatment groups. Water uptake for the two treatment methods, dip and spray, was calculated and the mean values were used to determine solution ai for other treatment groups.

For treatment groups 1-8, low concentration treatments, a target of 100 mg. ai/sqft was desirable. Based on water uptake of spray-treated control samples, a 3.6% ai solution was used for applying Si-Quat via spray application. For Si-Plus, the solution received from the manufacturer was a 2.0% ai solution, therefore it was applied as a 2.0% ai solution until a target weight was achieved, calculated to deliver the same 100 mg. ai/sqft. Based on water uptake of dip-treated control samples, a momentary dip in 0.51% ai solution was used for Si-Quat and Si-Plus application.

For treatment groups 9-16, high concentration treatments, a target of 150 mg. ai/sqft was desirable. Based on water uptake of spray-treated control samples, a 5.4% ai

solution was used for applying Si-Quat via spray application. The Si-Plus was applied by spray until a target weight was achieved as a 2.0% solution. Based on water uptake of dip-treated controls, a momentary dip in 0.76% ai solution was used for Si-Quat and Si-Plus application.

Treatment groups 17-20 were treated with the ready-to-use (RTU) product, Thompson's® Water Seal®, by dip and spray applications in accordance with label directions. For comparison of the efficacy of oven-curing silane-based treatments, treatment groups were paired, one oven-cured group, and one non oven-cured group per treatment used.

Testing

Once treated, test samples were re-conditioned at 60% RH and 75 °F (23.9 °C) until a constant weight was achieved prior to testing. The samples were then removed from the conditioning chamber and each treatment group (9 specimens) was weighed and then immersed in deionized water for 30 minutes. in accordance with AWPA E4-03. Immersed specimens were then removed from the deionized water, surface dried, and weighed to calculate the percent weight gain of each test specimen (deviation from the standard; tangential swelling was not evaluated). Percent weight gain results were recorded and mean values were analyzed by Tukey, a test for the significance of means.

Results and Discussion

For each of the four treatment methods, dip, dip oven-cured, spray, and spray oven-cured, the average percent weight gain for each treatment group was evaluated to determine any significant differences between treated test groups and untreated control

groups. As shown in Figures **4.2** – **4.5**, all treatments were significantly different than the untreated controls. As shown in Figure **4.2**, the average percent weight gain for the high concentration (150 mg. ai/sqft) Si-Plus spray (36%) was not significantly different from the Thompson's® Water Seal® spray (30%). The average percent weight gains of oven-cured silane-based spray treatments shown in Figure **4.3** were significantly different than the oven-cured Thompson's® Water Seal® spray treatment (31%). Average percent weight gains for oven-cured spray treatments (Figure **4.2**) were not significantly different than non oven-cured spray treatments (Figure **4.3**).

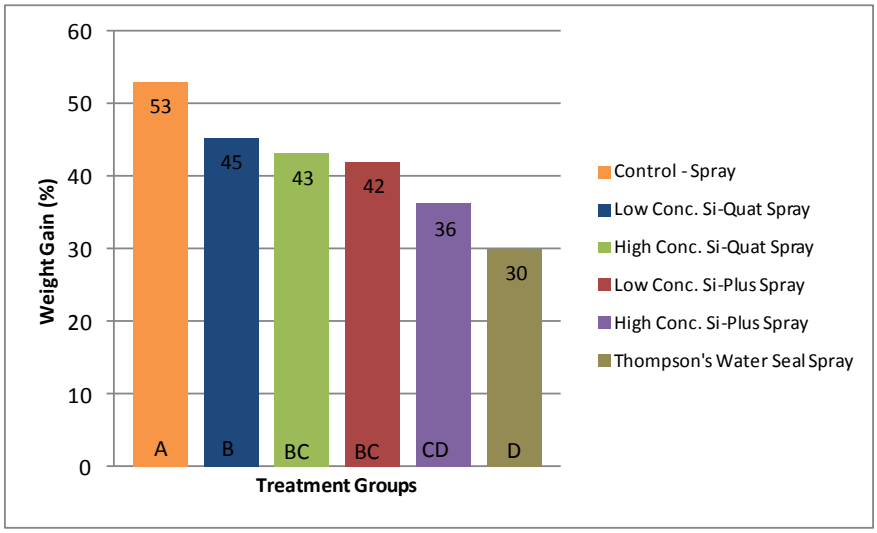

Figure 4.2

Average percent weight gain of spray-treated, non oven-cured treatment groups. Treatment groups with the same letter are not significantly different.

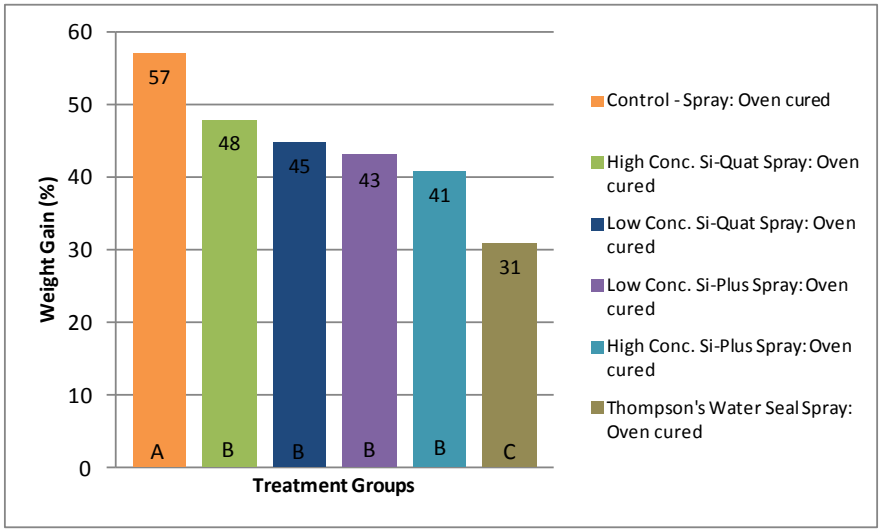

Figure 4.3

Average percent weight gain of spray-treated, oven-cured treatment groups. Treatment groups with the same letter are not significantly different.

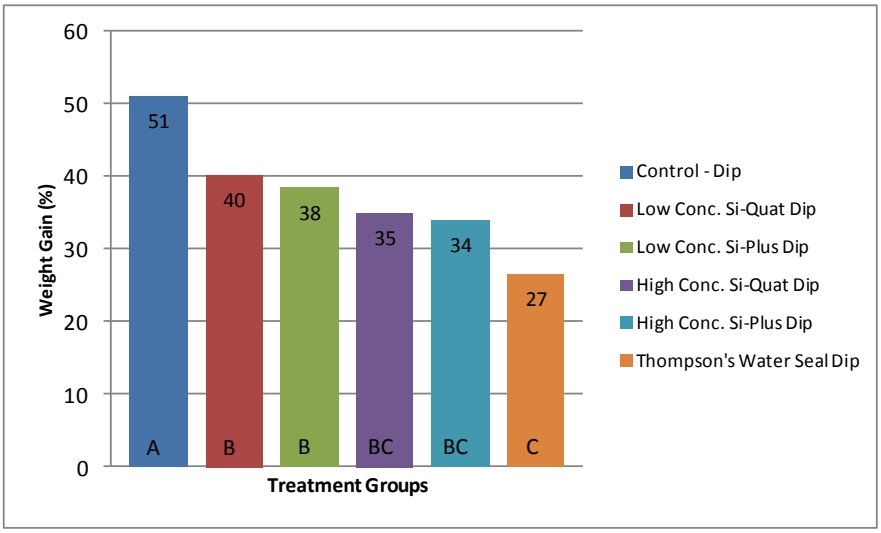

Figure 4.4

Average percent weight gain of dip-treated, non oven-cured treatment groups. Treatment groups with the same letter are not significantly different.

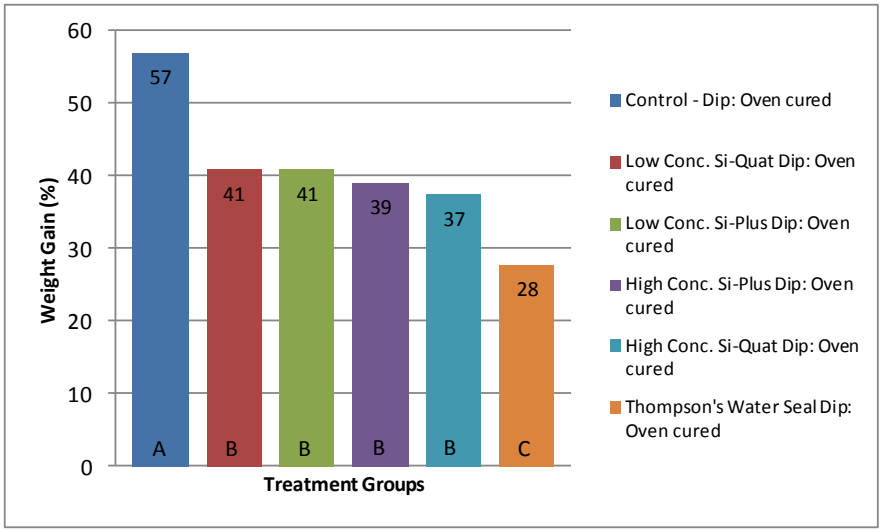

Figure 4.5

Average percent weight gain of dip-treated, oven-cured treatment groups. Treatment groups with the same letter are not significantly different.

As shown in Figure **4.4**, the average percent weight gains for the high concentration (150 mg. ai/sqft) Si-Quat dip treatment (35%) and the high concentration (150 mg. ai/sqft) Si-Plus dip treatment (34%) were not significantly different from the Thompson's® Water Seal® dip treatment (27%). The average percent weight gains of oven-cured silane-based dip treatments shown in Figure **4.5** were significantly different than the oven-cured Thompson's® Water Seal® dip treatment (28%). Average percent weight gains for non oven-cured dip treatments (Figure **4.4**) were not significantly different from oven-cured dip treatments (Figure **4.5**) with the exception of a single treatment group. The average percent weight gain for the high concentration (150 mg. ai/sqft) Si-Plus dip treatment (34%) was significantly different than when oven-cured (39%).

Conclusion and Recommendations

Results for this study on the efficacy of a the silane 3 (trimethoxysilyl) propyldimethyloctadecyl ammonium chloride as a water repellent on wood substrates applied under non-pressurized treatment methods, indicate that it significantly reduced the amount of weight gain (water uptake) by treated samples when immersed in water in comparison to untreated control samples. Results of this testing procedure support the efficacy of Thompson's® Water Seal®, a ready-to-use water repellant product labeled for use on wood materials. Three silane-based treatment groups, all treated with a high concentration (150 mg. ai/sqft), yielded results comparable to Thompson's® Water Seal®. Oven-curing of silane-based formulations once applied on test specimens had insignificant effects on treatment efficacy with the exception of a single treatment group. A possible trend is evident from the data collected in that higher mg ai/sqft of silane-based formulation on a wood substrate yielded a lower percent weight gain (water uptake). Further testing of this hypothesis should be conducted at possible treatment levels of 200 mg. ai/sqft and 250 mg. ai/sqft. Since the over-treatment of both high and low concentration Si-Quat spray treatment groups yielded a similar percent weight gain of 45, it is hypothesized that Si-Quat spray is not as effective as the Si-Quat dip or Si-Plus formulations, though further testing should be conducted.

CHAPTER V

CONCLUSIONS

The primary goal of this study in its entirety was to evaluate the organosilane 3-(trimethoxysilyl) propyldimethyloctadecyl ammonium chloride (Si-Quat) utilized on wood substrates. It is hypothesized that wood treatment with Si-Quat can supplement the phytosanitation of wood by providing residual moisture control and toxicity to organisms.

Results of experiments evaluating the residual efficacy of Si-Quat in conjunction with standardized heat treatment to inhibit mold growth on unseasoned lumber show that mold readily grew on unseasoned, heat treated control specimens but was retarded on specimens treated with the Si-Plus compound by 25% or greater. Experiments evaluating termite resistance to Si-Quat treated wafers resulted in increased termite mortality and decreased feeding when compared to control wafers. Results of water repellency tests indicated that both Si-Quat and Si-Plus significantly reduce the percent weight gain of water by treated test specimens. Though further research is needed to provide specific treatment levels and application methods of the silane-based formulations utilized in this study as well as toxic thresholds, perhaps wood treatments with Si-Plus could be considered for supplementation to standardized phytosanitation measures.

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